THE INFLUENCE OF LIGHTWEIGHT FINES SUBSTITUTED FOR CONVENTIONAL FINES ON FREEZE-THAW DURABILITY OF PORTLAND CEMENT CONCRETE

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by

D.L. GRESS

Joint Highway Research Project

PURDUE UNIVERSITY
LAFAYETTE INDIANA

Final Report

THE INFLUENCE OF LIGHTWEIGHT FINES SUBSTITUTED
FOR CONVENTIONAL FINES ON FREEZE-THAW DURABILITY
OF PORTLAND CEMENT CONCRETE

To: J. F. McLaughlin, Director
Joint Highway Research Project

From: H. L. Michael, Associate Director
Joint Highway Research Project

September 12, 1968

Project No.: C-36-37BB

File No.: 5-8-28

The attached Final Report is submitted for the record and for information. It is titled "The Influence of Lightweight Fines Substituted for Conventional Fines on Freeze-Thaw Durability of Portland Cement Concrete" and has been authored by Mr. David L. Gress, Graduate Assistant on our staff. The report is of research conducted under a Plan of Study approved by the Board on December 17, 1966. Professor C. F. Scholer directed the study. Mr. Gress also used the report as his thesis as partial fulfillment of the requirements for the MSCE degree.

The research found that the use of expanded shale fine aggregates in place of conventional fines did not improve the scaling resistance of concrete.

The report is presented to the Board for the record. Comments and review are invited and would be appreciated.

Respectfully submitted,

Harold L. Michael

Harold L. Michael
Associate Director

HLM:mz

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Project: C-36-37BB
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September 12, 1968

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TABLE OF CONTENTS

TABLE OF CONTENTS (Con'd.)

TABLE OF CONTENTS (Con'd.)

LIST OF TABLES

LIST OF TABLES (Con'd.)

LIST OF FIGURES

LIST OF FIGURES (Con'd.)

LIST OF FIGURES (Con'd.)

ABSTRACT

Gress, David Lee, M.S.C.E, Purdue University, June 1968. The Influence of Lightweight Fines Substituted for Conventional Fines on Freeze-Thaw Durability of Portland Cement Concrete. Major Professor: Charles F. Scholer.

This investigation was undertaken to identify and quantify those factors which reduce freeze-thaw scaling when lightweight fines are substituted for the conventional sand in portland cement concrete. This was based on the hypothesis that the lightweight fines would produce a durable concrete. A model was employed which would have offered many benefits if the hypothesis had been proven. Normal weight crushed stone was used for the coarse aggregate. A local sand of glacial origin and proven durability was used for comparison purposes in control mixes.

Two methods of investigating concrete freeze-thaw durability were conducted. One of these, ASTM Designation: C 291-61T, did not evaluate the potential scaling resistance of the concretes. The other test, a scaling resistance test which consisted of freezing pure water on the concrete surface and thawing with calcium chloride, showed a difference among all three aggregates.

A "solid bubble" hypothesis by which porous fines might act as an air bubble was proposed.

An investigation of the porosity of the two lightweight aggregates showed a difference in the effective porosities. The coated aggregate had a higher effective porosity and a larger amount of pores smaller than 5 microns than the non-coated aggregate.

The use of expanded shale fine aggregates substituted for the conventional fines in concrete did not improve the scaling resistance of concrete.

INTRODUCTION

A practice of applying salts to concrete pavement to remove ice was initiated around 1930. This practice has aggravated the freeze-thaw problem. A new type of surface scaling uncommon to the regular freeze-thaw problem accompanied the new use of salt. A field survey clearly disclosed that the surface scaling resulted from the application of salt and not by any change in the characteristics of the cement or the quality of the concrete (1)*.

Studies of air-entrained concrete were initiated in the late 1930's. These studies were part of a comprehensive program of research to find a means of preventing the surface scaling when salts or granular material mixed with salts are applied for ice removal (1).

Air-entrained concrete, now universely used, helps the freeze-thaw and scaling problem. However, as disclosed in recent bridge deck surveys (2,3), many bridge decks do not have complete protection. A number of factors may contribute to the problem. Bridge decks are subject to freezing from

*Numbers in parentheses refer to references listed in the List of References.

two surfaces. Construction procedures may result in surfaces having reduced durability due to the air-entrainment being destroyed. Lack of uniformity and overworking contribute to the difficulties (4). The frequent applications of de-icing salts on the surface of the bridge decks is another important reason (5). Poor drainage of the pavement surface aggravates the problem.

This increased durability obtained by the use of entrained air can be achieved only if the proper amount of air is entrained in the concrete. A concrete will not have the desired degree of durability if it is produced with an insufficient amount of entrained air or if the distribution of the entrained air bubble is lost.

Recent surveys (1,2,3,4) have indicated bridge decks which showed deterioration from freezing and thawing action were usually constructed of insufficiently air-entrained concrete.

If concrete contains the proper amount of entrained air, it is felt it will withstand the effects of freezing and thawing action indefinitely. The problem that exists is to produce a concrete with the required amount of entrained air. If a conventional air-entrained bubble could be replaced by a "solid bubble", the problem mentioned above could be

easily solved. Theoretically this "solid bubble" could be a void or a system of voids where each individual void is encased by sound, durable material. In reality, this "solid bubble" may be nothing more than a highly porous lightweight aggregate. It could have advantages over normally entrained air because it could not be beaten out of the concrete as can a regular bubble.

Lightweight fines have been substituted for the conventional fines in concrete (6) and the results of the durability showed definite promise. The fact that this method did show promise led this investigator to consider the problem in more detail.

REVIEW OF LITERATURE

The New York State Thruway Authority recently instituted an exposure test of concrete slabs for the expressed purpose of finding materials that would give the maximum service in the maintenance repairs of structural concrete and to eliminate materials of marginal durability (6). One factor considered was the use of a certain expanded shale aggregate substituted for the conventional sand in portland cement concrete. It should be borne in mind that this was a field usage test devised and conducted by maintenance forces.

The testing procedure consisted of subjecting 18"x18"x3" slabs with a 2"x3/4" coping around the top to atmospheric conditions. Every morning that the water was frozen, a mixture of fifty percent sodium chloride and fifty percent calcium chloride, in sufficient quantity to produce a five percent salt solution, was applied to induce thawing. This cycle was followed for five days after which time the slabs were flushed clean and refilled with clear water and left for forty-eight hours subject only to temperature induced freeze-thaws. The de-icing salts were then applied again for

five cycles. Thus, during every five-day period, the slabs were either going through freeze-thaw cycles or lay covered with the brine solution. The slabs were flushed clean and left to weather in an exposed position during the Spring, Summer and early Fall.

It was concluded that the absence of scientific instrumentation and precise controls left unanswered a number of questions that could be raised. It was found, however, that by the use of expanded shale fine aggregate, a concrete could withstand innumerable freeze-thaw cycles and applications of de-icing chemicals without evidencing distress.

Numerous durability tests have been performed on complete lightweight concrete (8,9,10) as there have been on sand replacement lightweight concrete (8,11,12) but only the New York Thruway Authority have conducted tests on concrete made with expanded shale fine aggregate.

In a recent investigation (8) scaling tests were conducted on concrete slabs. One phase of the test was to determine the effect of using a manufactured lightweight aggregate on the resistance of concrete to scaling. Slabs were prepared with concrete in which the lightweight fine and coarse aggregates were used, and also where lightweight coarse aggregates were used in combination with a natural fine

aggregate (sand replacement). These slabs were subjected to thawing with a de-icer. Three of the four slabs prepared with lightweight aggregates developed a form of surface map cracking with the more severe condition occurring when only the coarse aggregate fraction was the lightweight material. On the basis of their ratings, the scale resistance of the lightweight aggregate concrete was slightly better than that of the normal weight concrete used in their control slabs.

One source (7) reported that a series of tests were conducted to evaluate expanded aggregates for use in skid resistant pavement surfaces. One phase of this testing was to substitute expanded shale fine aggregate for the conventional limestone fines in portland cement concrete. Laboratory and field experiments performed on these lightweight expanded shale surfaces indicated large increases in the skid resistance over surfaces constructed of limestone aggregates. The increase in skid resistance was almost directly proportional to the amount of expanded shale aggregate used.

Data were unavailable at the time of printing to evaluate the comparative durability of the expanded shale mix. No defects in the integrity of an experimental pavement surface had been observed after nearly four years of service.

PURPOSE AND SCOPE

This study is concerned with the effect of freeze-thaw action on concrete made with two expanded shale lightweight fine aggregates with a conventional coarse aggregate.

The purpose of this study was to investigate the durability of concrete made with two lightweight fine aggregates when subjected to freeze-thaw action.

MATERIALS

A general description of the materials used in this investigation is presented.

Coarse Aggregate

A good durable limestone aggregate from the Ste. Genevieve formation containing no chert was used for the coarse aggregate fraction. This aggregate is designated 53-2S in the Joint Highway Research Project Concrete Laboratory at Purdue University and will hereafter be referred to by that designation. Table 1 presents the physical properties of this aggregate.

Fine Aggregate

Three aggregates were used for the fine fraction of the concrete mixes, one of which was a control sand used only for relative comparison purposes. The two lightweight fine aggregates were manufactured in accordance with the following ASTM specifications: C 330-64T, Tentative Specifications for Lightweight Aggregates for Structural Concrete, C 331-64T, Tentative Specifications for Lightweight Aggregates for

Concrete Masonry Units, and C 332-61, Specifications for Lightweight Aggregates for Insulating Concrete.

Lightweight Coated Aggregate

One of the lightweight fine aggregates was a coated expanded shale produced by the rotary kiln process. The raw shale was crushed and graded prior to introduction into the kilns, where it was expanded as individual particles at temperatures in excess of 2000 degrees Fahrenheit. A photomicrograph of the No. 16 size fraction for this aggregate is presented in Figure 1.

Lightweight Noncoated Aggregate

The other lightweight fine aggregate was a noncoated expanded shale produced by the rotary kiln process. The raw shale was crushed to the proper size and introduced to the rotary kiln, where it was expanded into a vitreous material referred to as the clinker at a temperature of approximately 2200 degrees Fahrenheit. The clinker was then cooled, crushed, and screened to the proper gradations. A photomicrograph of the No. 16 size fraction for this aggregate is presented in Figure 1.

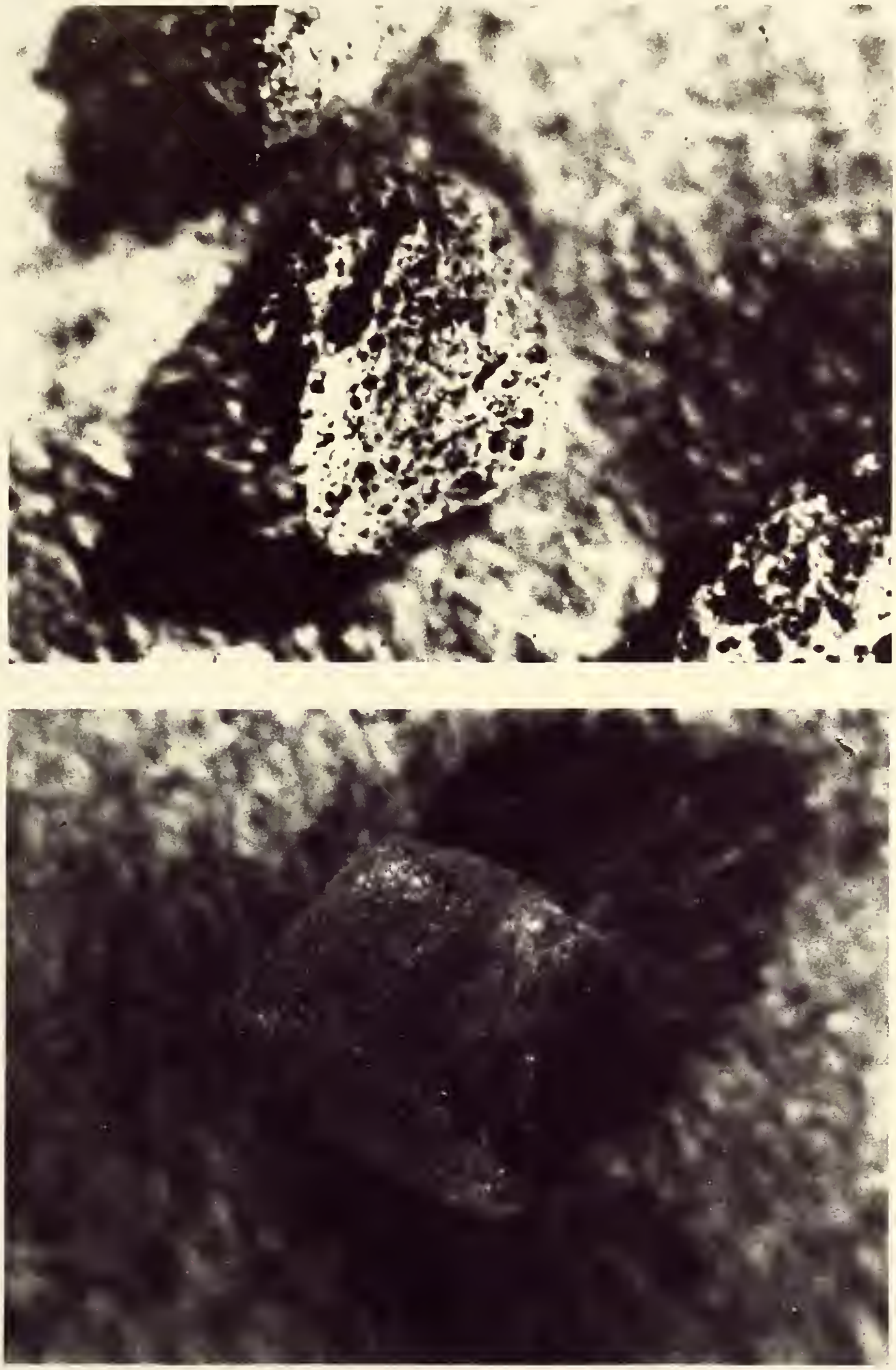


FIGURE 1 PHOTOMICROGRAPH PICTURES OF THE NONCOATED (TOP) AND THE COATED AGGREGATE OF THE NO. 16 SIZE FRACTION

Control Sand

The control sand was a local sand obtained from a river terrace deposit of glacial origin. This fine aggregate was designated 79-1G sand in the Joint Highway Research Project Concrete Laboratory at Purdue University and will hereafter be referred to by that designation. Refer to Table 2 for the physical properties of 79-1G.

Cement

A Type I portland cement from a single clinker batch was used in all of the mixes. The physical and chemical properties of this cement are listed in Table 3.

Air-Entraining Agent

The air-entraining agent used was a neutralized vinsol-resin solution which met the specifications of ASTM C 260-65T, Tentative Specifications for Air-Entraining Admixtures for Concrete.

De-Icer

The de-icer used was regular flake calcium chloride, Type 1 which met the specifications of ASTM D 98-59, Standard Specifications for Calcium Chloride.

TABLE 1

PHYSICAL PROPERTIES OF 53-2S

Bulk Specific Gravity	2.68
True Specific Gravity	2.70
Absorption	1.0%

Gradation

Size	Percent Passing
1"	100
3/4"	90
1/2"	40
3/8"	7
No. 4	0

Dry Rodded Weight 104.4 lbs. per cubic foot

TABLE 2

PHYSICAL PROPERTIES OF 79-1G

True Specific Gravity	2.70
Bulk Specific Gravity	2.58
Bulk Specific Gravity (Saturated Surface Dry)	2.62
Apparent Specific Gravity	2.67
Absorption	1.53%

TABLE 3

PHYSICAL AND CHEMICAL PROPERTIES OF CEMENT*

Physical Properties

Fineness, No. 325 Sieve	95.9 percent
Specific Surface, Blaine	3380 sq. cm. per gm.
Initial Set	3 hrs., 15 min.
Final Set	5 hrs., 5 min.

Chemical Analysis

Compound	Percent Present
Silicon dioxide, SiO_2	21.76
Aluminum oxide, Al_2O_3	5.41
Ferric oxide, Fe_2O_3	1.97
Calcium oxide, CaO	65.30
Magnesium oxide, MgO	1.11
Sulphur trioxide, SO_3	2.43
Loss on Ignition	1.78

*The portland cement is designated No. 317 in the Joint Highway Research Project Concrete Laboratory at Purdue University.

TABLE 3 (Con'd.)

Calculated Compound Composition

Compound	Percent Present
Tricalcium silicate, C_3S	51.20
Dicalcium silicate, C_2S	23.83
Tricalcium aluminate, C_3A	11.00
Tetracalcium aluminoferrite, C_4AF	5.99
Calcium Sulphate, $CaSO_4 \cdot 2H_2O$	4.13

EQUIPMENT

A general description of the equipment used in this investigation is presented.

Freezer

A large walk-in freezer was employed for the scaling test. The freezer contained shelves for storing the pads. A constant temperature of zero degrees Fahrenheit was maintained through the freezer by means of a large fan located behind a system of cooling coils. Cooling coils were also located under the shelves.

Mixer

A tub type mixer capable of mixing up to one and one-half cubic foot of concrete was used for all of the concrete mixing. The apparatus was a Lancaster Counter Current Batch Mixer, manufactured by Posey Iron Works, Inc. The paddles revolved around the tub approximately fifty times per minute.

Vibrating Table

A vibrating table capable of vibrating loads up to fifteen pounds was used. The vibrating table was a model-PJ15,

manufactured by the Syntron Company. This model had a variable amplitude with a frequency of 60 cycles per second.

Mercury Porosimeter

An Aminco-Winslow Mercury Intrusion Porosimeter produced by the American Instrument Company was used and operated up to pressures of 15,000 psi. A unique filling device was used in conjunction with this porosimeter to extend the pore size range of testing. This filling device allowed the penetrometer tube and sample to be evacuated, filled with mercury and tested up to one atmosphere while it remained in a horizontal position. This extended the pore size range of testing by preventing any de-absorbing of the mercury from the pores due to a negative head normally encountered in the tilt type filling device.

PROCEDURES

Procedures used for the experimental design, mix design, batching, mixing and placing, and testing are presented. These procedures were used throughout the extent of this investigation.

Experimental Design

The experiment was designed with four variables, lightweight fine aggregate, gradation of the fine aggregate, water cement ratio and volume of fine aggregate. A complete listing of the factors and their levels is presented in Table 4.

There are 2x4x4x4 = 128 combinations for the proposed model. Supply of materials and time limited the number of combinations that could be produced. Therefore, a fractional factorial design was used. Half replication was employed with eight repeats chosen randomly from the 128/2 or 64 combinations. Thus, a total of 72 treatment combinations were produced for the experiment. Appendix I contains the statistical details of the experimental design and analysis.

TABLE 4

FACTOR AND LEVEL DESCRIPTION

FACTOR	FACTOR DESCRIPTION	LEVEL	LEVEL DESCRIPTION
A	Lightweight Fine Aggregate	1	Noncoated Lightweight Aggregate
		2	Coated Lightweight Aggregate
B	Gradation of Fine Aggregate	1	FM = 2.35
		2	FM = 2.60
		3	FM = 2.85
		4	FM = 3.10
C	Water/Cement Ratio	1	W/C = 0.30
		2	W/C = 0.35
		3	W/C = 0.40
		4	W/C = 0.45
D	Volume of Fine Aggregate	1	Vol. Fines = 55.55%
		2	Vol. Fines = 57.32%
		3	Vol. Fines = 62.50%
		4	Vol. Fines = 65.22%

Eight control mixes were made to compare with the two lightweight fine aggregate concrete. These mixes were made with 79-1G control sand and shall hereafter be referred to as the control sand concrete. The control sand concrete was designed by using all possible combinations of the first and fourth levels of the three factors B, C and D. The control sand concrete consisted of mix numbers 73 through 80. The level combinations employed are located in Appendix II. The results of the control sand concrete could not be analyzed statistically with the above model due to unknown statistical confounding, but by means of direct comparison, the control sand concrete was compared with the lightweight fine aggregate concrete.

Mix Design

Numerous reports concerning mix design methods for structural lightweight concrete exist in the literature (13, 14,15,16). However, as mentioned in the literature review, little has been reported on concrete made with normal weight coarse aggregate with lightweight fines substituted for the conventional fines. Many of the problems encountered in the mix design of lightweight sand concrete are common to those of structural lightweight concrete. These problems are associated with characteristics of the lightweight aggregates

and require special consideration when designing a mix (13). The aggregates are sharp and angular throughout the range of sizes, even particles passing a No. 200 sieve. This tends to make concrete harsh and less workable just as crushed rock makes a concrete more harsh than rounded sand and gravel. Another characteristic of lightweight aggregates that requires special consideration is the variable unit dry weight that exists among the different sizes. The aggregates are highly absorptive. To further complicate the problem, the rate of absorption is variable, thus making it impractical to utilize specific gravity values in mix design and batch proportioning.

Volume Batching

As mentioned above, one of the characteristics which must be considered is the variable unit dry weight that exists among the different sizes. Generally, this unit dry weight varies inversely with the size fraction.

For conventional aggregates the bulk specific gravities of materials retained on the different sieve sizes are nearly equal. Therefore, the fineness modulus, on a weight basis, gives a true indication of the volumes occupied by each size fraction. The bulk specific gravity of the various size fractions of lightweight aggregate increases as the particle size decreases. Hence, some coarse sized particles might

float in water while the particles finer than the No. 100 sieve have a specific gravity approaching that of conventional sand and gravel. The total volume occupied by each size fraction and not the weight of material retained on each sieve determines the final void content, paste content and workability of the concrete. Table 5 shows an example of the fineness modulus by weight and by volume for a lightweight aggregate (16). The fineness modulus by volume, of 3.23, had a considerably coarser grading than that normally associated with the fineness modulus of 3.02 by weight. Therefore, on a weight basis, lightweight aggregates require a larger percentage of material retained on the finer sieve sizes than do the heavier aggregates to provide an equal volume (16).

Two of the four variables included in the experiment were gradation and volume of fine aggregate. Due to the problems previously mentioned regarding the bulk specific gravity, the lightweight fine aggregates were proportioned on a volume basis rather than by weight.

It has been reported (17) that good reproducible results of specific gravity for lightweight aggregates can be obtained by using a pycnometer.

TABLE 5

FINENESS MODULUS BY WEIGHT VERSUS BY VOLUME

Sieve Size	Percent Retained by Weight	Cumulative Percent Retained by Weight	Specific Gravity	Percent Retained by Volume	Cumulative Percent Retained by Volume
4	0	-	1.40	-	-
8	21.6	21.6	1.55	25.9	25.9
16	24.4	46.0	1.78	25.4	51.3
30	18.9	64.9	1.90	18.5	69.8
50	14.0	78.9	2.01	13.0	82.8
100	11.6	90.5	2.16	9.9	92.7
PAN	9.5	100.0	2.40	7.3	100.0

Fineness Modulus (by weight) = 3.02

Fineness Modulus (by volume) = 3.23

The fine aggregates were dried and separated using the following U.S. Standard sieves, sizes 3/8", No.'s 4, 8, 16, 30, 50, 100, and 200. True specific gravity tests were run on each of the material fractions below the No. 4 size by means of a pycnometer. Figure 2 shows the relation between specific gravity and size fractions for the two lightweight aggregates. A definite difference existed among the size fractions thus confirming that which was reported in the literature.

A volume batching procedure was developed in order to control the proportioning of the mixes. A "dry vibrated density" was determined by a procedure in which the densities of the various material fractions could be reproduced to 0.01 grams per cubic centimeter of the original. The procedure consisted of vibrating each size fraction in a known volume container on a vibrating table until a constant weight was obtained. This weight divided by the volume of the container produced the "dry vibrated density". It should be noted that great care was taken to reproduce these densities. After many failures the most successful technique was to fill the container with the material fractions by means of a funnel in a rotating fashion. The aggregate was not allowed to fall from the funnel into the container. Instead, a height was maintained such that a cone of aggregate was

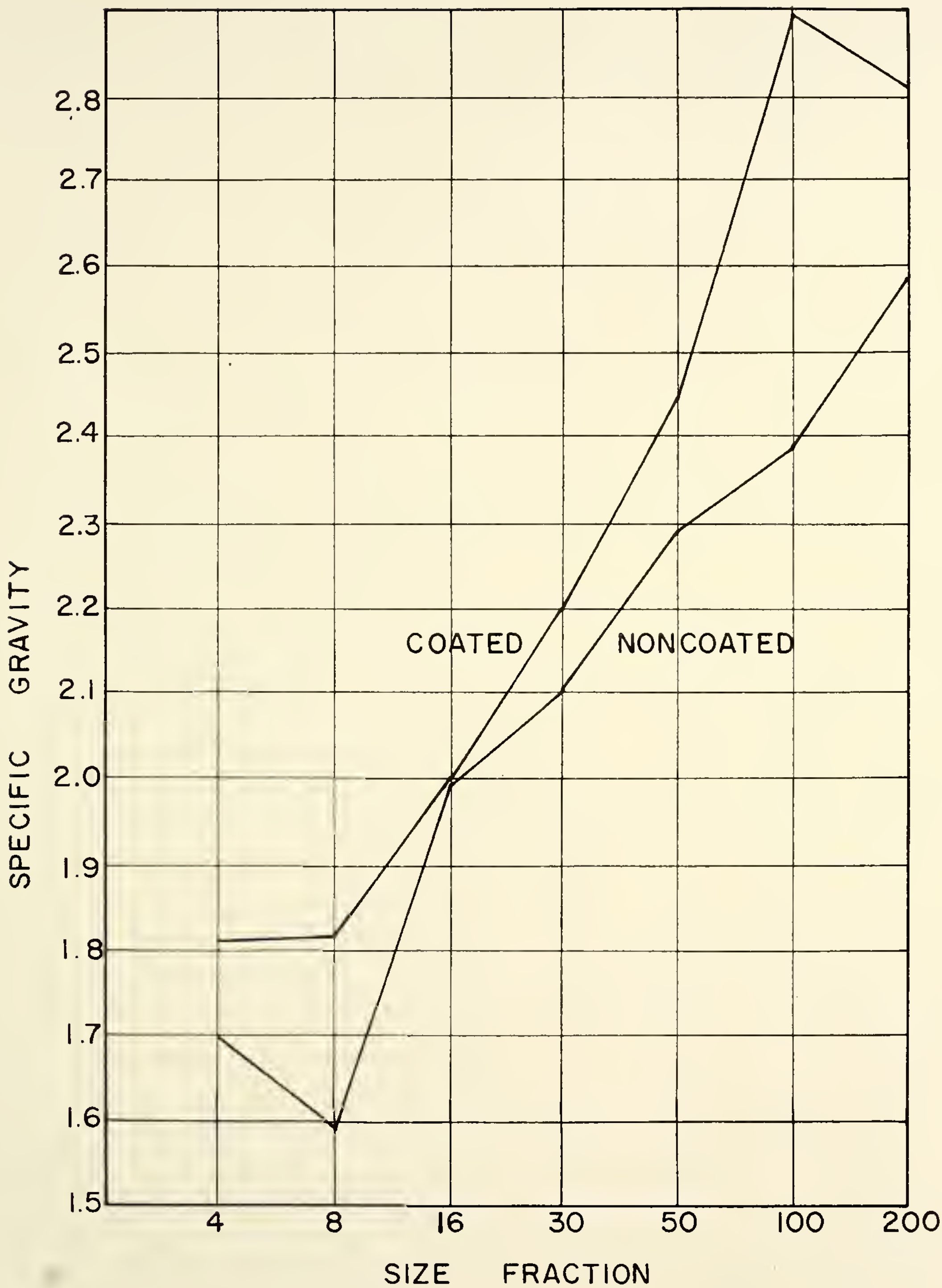


FIGURE 2 SPECIFIC GRAVITY VS. SIZE FRACTION FOR THE COATED AND NONCOATED AGGREGATE

constantly flowing from the funnel into the container. This procedure of filling produced a very loose packing of the particles.

Vibration was applied, after the container was filled, by means of a vibration table. The vibration table, as described under Equipment had a fixed frequency of 60 cycles per second and a variable amplitude. It was found that when the amplitude was increased, the energy applied to the system was increased and at some point the particles on the surface having no restraint from above had a tendency to jump out or to relocate themselves. This produced a density which varied spastically with time and was impossible to reproduce. Thus, the amplitude was adjusted so that the energy input was less than that required to cause any of the particules to jump free of the surface or to randomly relocate themselves.

At various time intervals the container was struck off with a straight edge and weighed. Vibration was continued until the weight increase was less than that required to increase the "dry vibrated density" by 0.01g/cc. This usually took at least 60-minutes of vibration to produce a constant density.

Each density was reproduced within 0.01g/cc and the average of the two final readings was used to produce the

"dry vibrated density". Figure 3 shows the relation between size fraction and "dry vibrated density" for the two lightweight aggregates used. Figure 4 shows the relation between size fraction and "dry vibrated density" for the control sand 79-1G.

Absorption

Lightweight aggregates are highly absorptive and both the rate and amount of absorption varies from one aggregate to another. This gives rise to the principal difficulty in proportioning by absolute volume procedures.

Various methods have been proposed for the determination of absorption of lightweight aggregates. The Saxer (18) direct method of absorption was tried with little success. The method itself is valid for coarse size lightweight but the results are not applicable for determining the total absorption of lightweight fine aggregates. The conventional method of determining absorption does not apply because it is impossible to obtain a saturated surface dry condition on the small particles of the aggregates.

A test was originated which would give some indication of the water required for mix design purposes. The test was not meant to give the absorption of the aggregate but the amount of water required to "wet" it. The water required

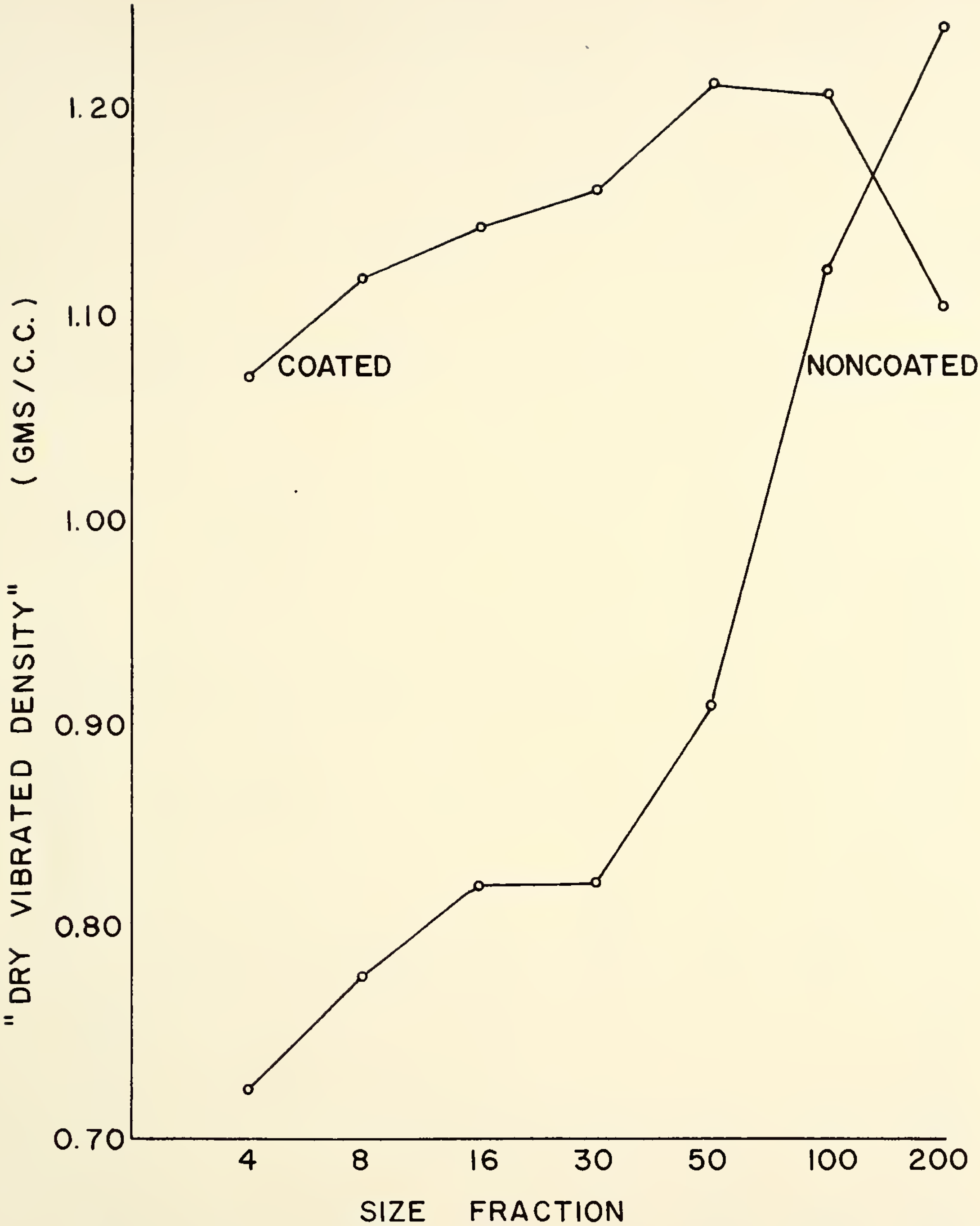


FIGURE 3 "DRY VIBRATED DENSITY" VS. SIZE FRACTION FOR THE COATED AND NONCOATED AGGREGATE

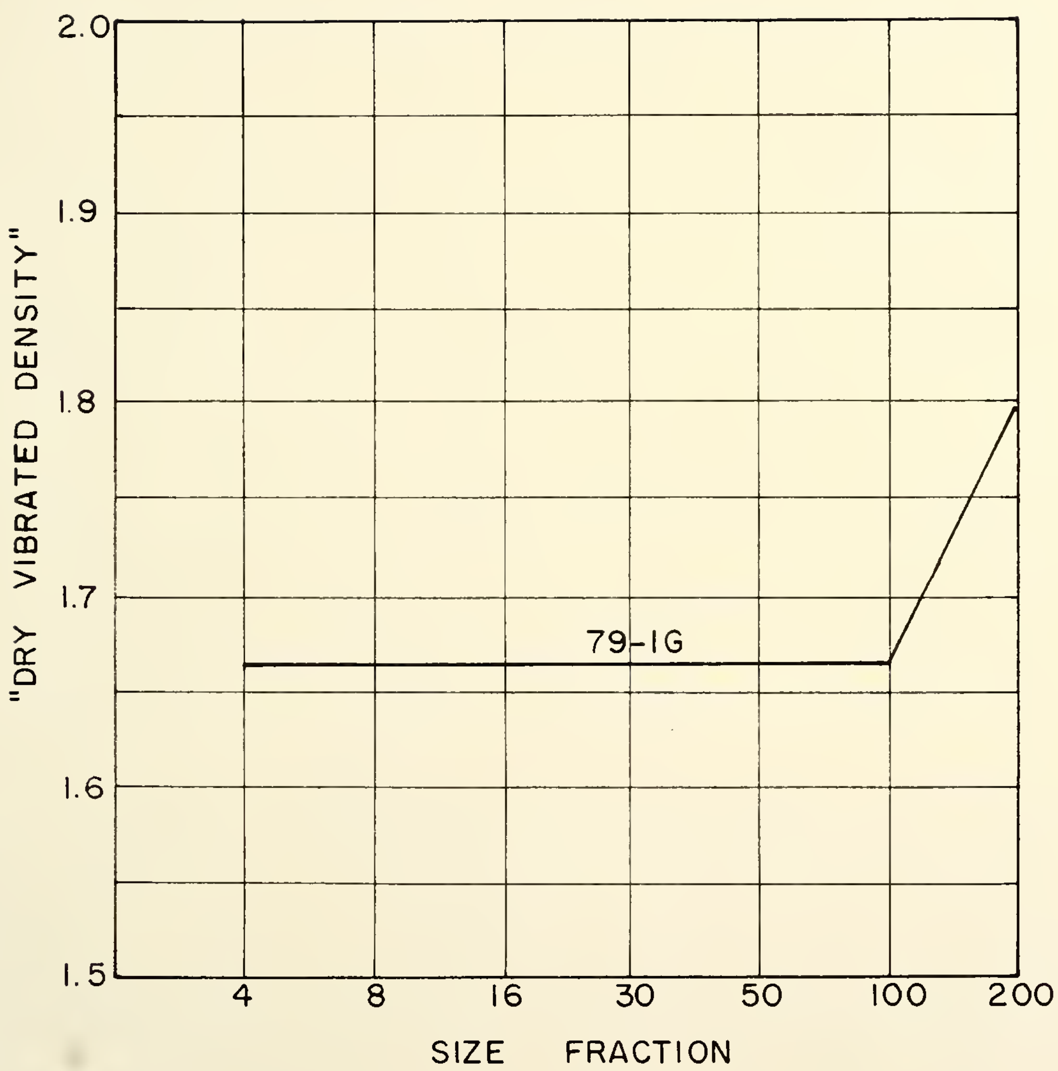


FIGURE 4 "DRY VIBRATED DENSITY" VS. SIZE FRACTION FOR 79-1G SAND

to "wet" the aggregate was expressed in percent of its dry weight.

The four gradations used in the experiment were batched by the "dry vibrated density" method for each of the aggregate types. The 500cc volumes were then wrapped in 12" square cotton cloth and submerged in water for 30-minutes. At the end of 30-minutes the cloth was spread over a No. 8 screen and the wet sample of aggregate was evenly distributed by means of a rod and allowed to drain for 30-minutes. The aggregate sample was then immediately removed from the cloth and weighed. Thus, an indication of the amount of water required was obtained for the four gradations of the fine aggregates. Figures 5, 6 and 7 show the relation between the water required to "wet" the aggregate versus the four gradations for the noncoated, coated and control aggregates, respectively.

Through experimentation in the laboratory, it was found that the amount of water required for the dry aggregate to produce a concrete of the proper consistency was approximately forty percent the values found in the test. Therefore, forty percent of these values determined by the water to "wet" the aggregate test were used for the water requirement of the aggregates in the mix design.

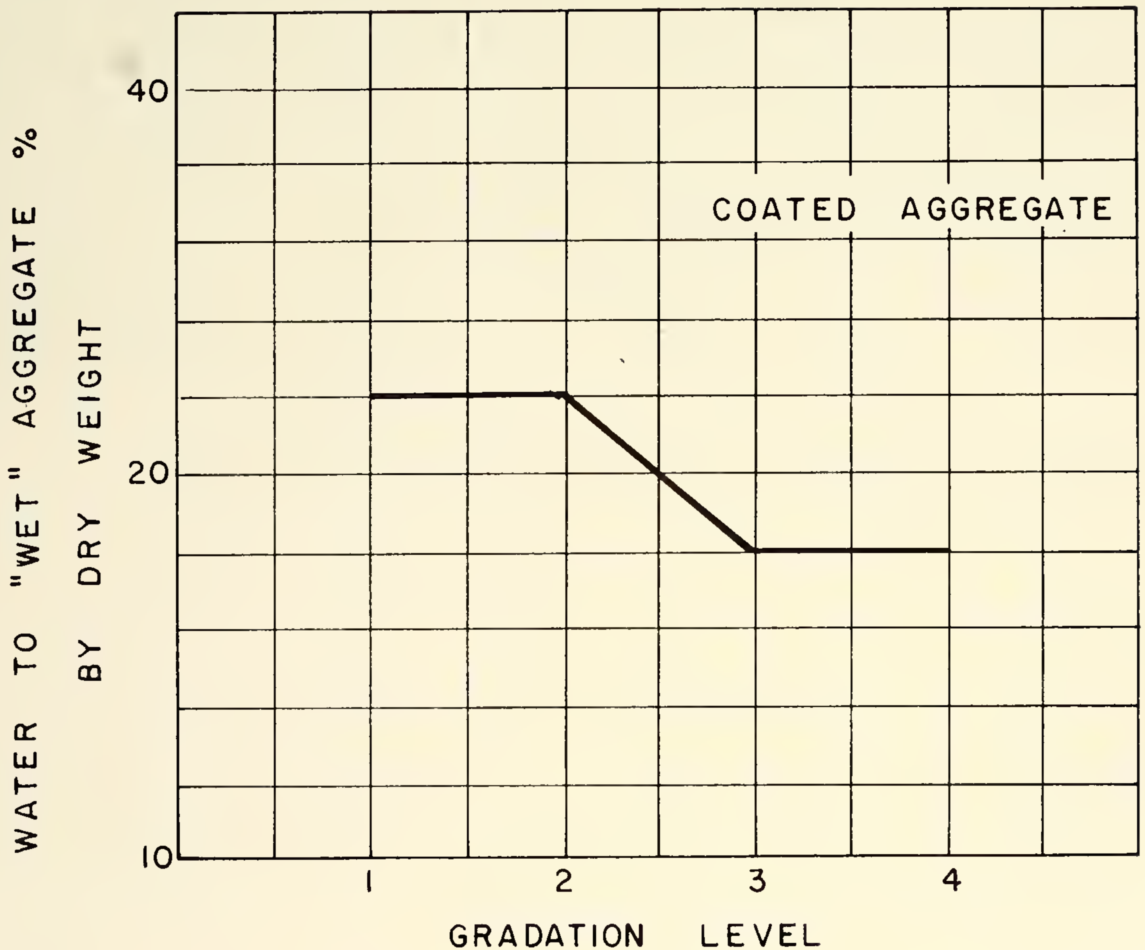


FIGURE 5 WATER TO "WET" AGGREGATE % BY DRY WEIGHT VS. GRADATION LEVEL FOR THE COATED AGGREGATE

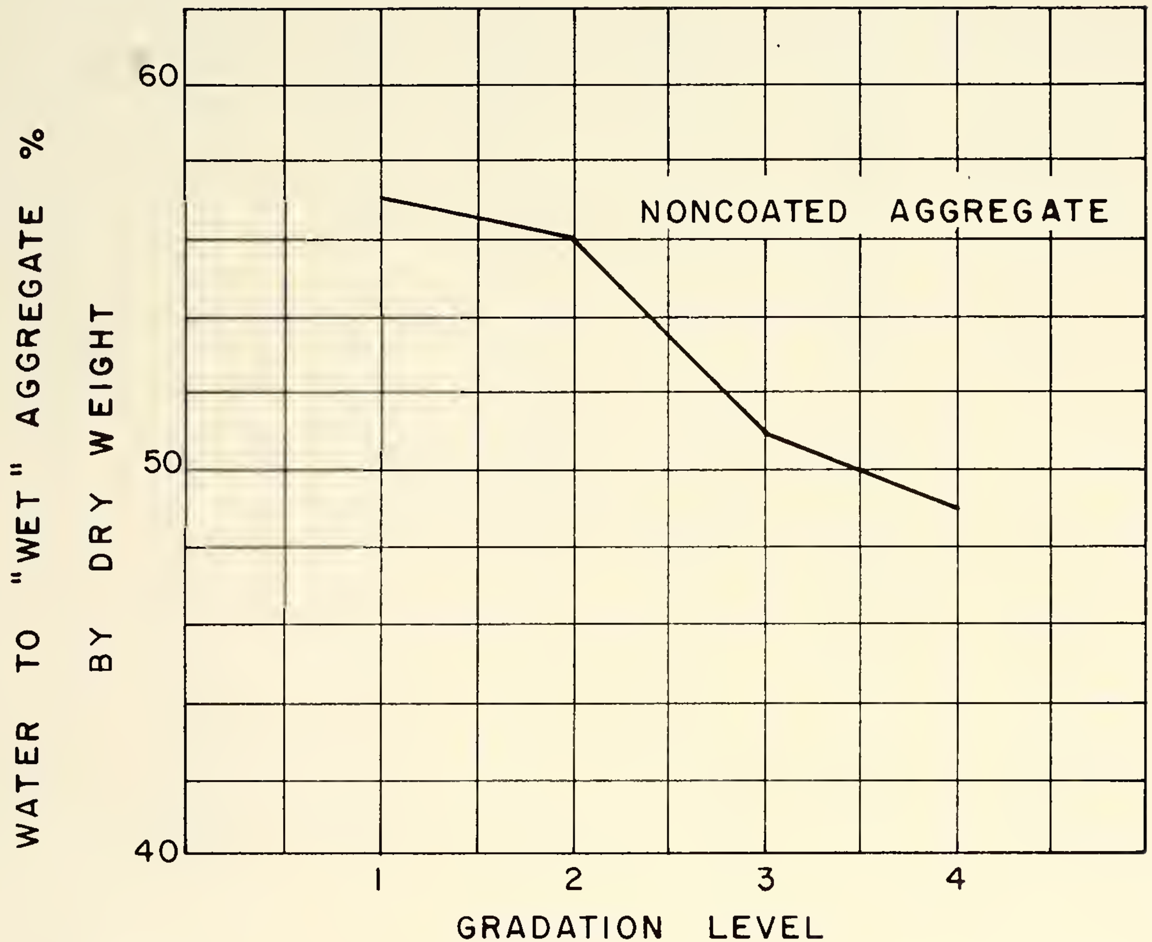


FIGURE 6 WATER TO "WET" AGGREGATE % BY DRY WEIGHT VS. GRADATION LEVEL FOR THE NONCOATED AGGREGATE

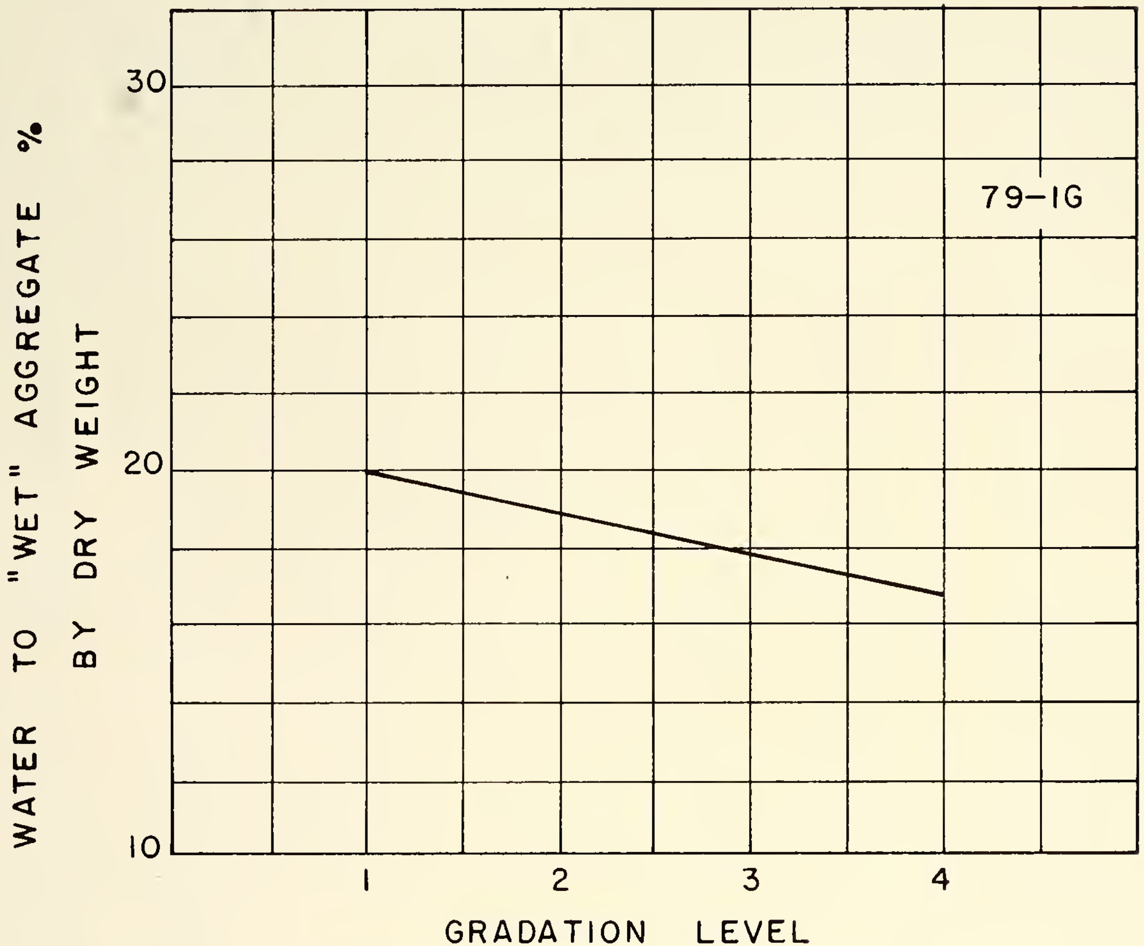


FIGURE 7 WATER TO "WET" AGGREGATE % BY DRY WEIGHT VS. GRADATION LEVEL FOR 79—1G SAND

Proportions

It has been reported that a good estimate of the proportions of fine and coarse aggregate for lightweight concrete may be obtained by the maximum density method (16). This procedure generally indicates that, for 3/4" maximum size aggregate, material passing the No. 4 sieve should be between forty and sixty percent of the total aggregate based on dry loose volume. Most published information (13, 15, 16, 17) on fine-to-coarse aggregate proportioning indicates that approximately forty to sixty-five percent by volume of the total aggregate should pass the No. 4 sieve. It must be noted that all of the literature involved complete lightweight aggregate and did not correspond to the requirements of only lightweight sand concrete but, for the purpose of this investigation, it was assumed the requirements were the same.

Particle Shape

The sharp, angular nature of crushed expanded shales calls for a higher percentage of fines passing the No. 4 sieve than the more rounded coated expanded shales and conventional sands (16).

Measurements of the long and short dimensions of particle slices from the two expanded shales were made. The different size fractions were mixed separately with white

cement and water, slices were cut from the hardened mortar, and thin sections made. The long and short dimensions of the particles exposed in the sections were measured and the ℓ/s ratios were calculated. The particles down to the No. 50 sieve size from both types of lightweight aggregates were tested. The ℓ/s ratio gave a relative indication of the particle shape.

The statistical analysis of the data is shown in Table A7 located in Appendix II. It was felt that the small difference of 1.8 vs. 1.6 for the average ℓ/s ratios of the non-coated and coated fine aggregates, respectively, did not merit adjusting the fines-to-coarse aggregate ratio.

Gradation and Volume Levels

Gradation and volume of the fine aggregate discussed in Experimental Design were two of the four variables in this experiment. Each of these variables or factors employed four levels in the factorial model. The levels of the gradation factor met the requirements of lightweight fine aggregates set forth in ASTM C 330-64T, Tentative Specifications for Lightweight Aggregate for Structural Concrete. Figure 8 shows the relation between accumulated percentages retained and size fraction for the four gradation levels.

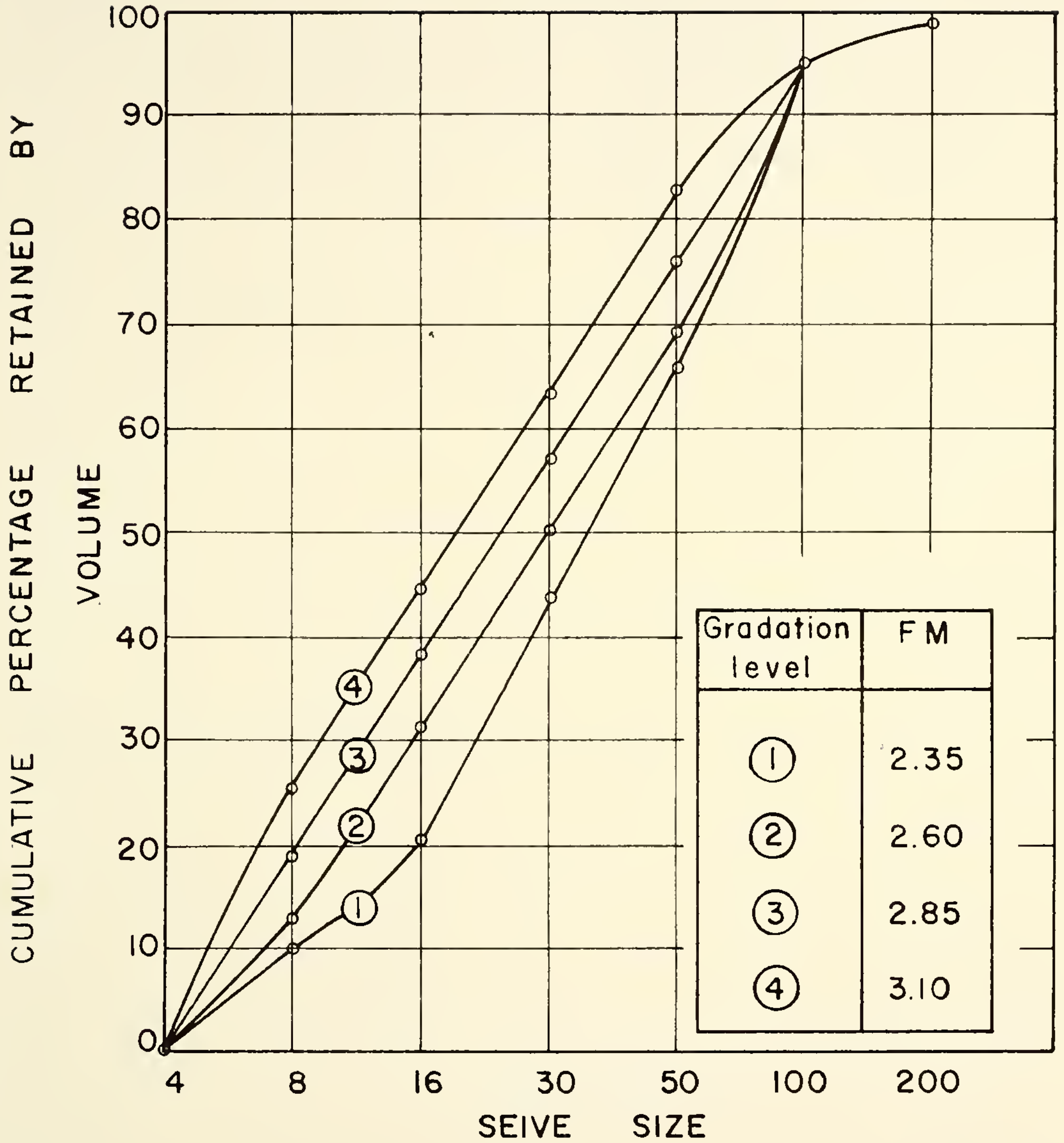


FIGURE 8 CUMULATIVE PERCENTAGE RETAINED BY VOLUME VS. SIEVE SIZE OF THE FOUR GRADATION LEVELS

Most published information on fine to coarse aggregate proportioning indicates that approximately forty to sixty-five percent by volume of the total aggregate should pass the No. 4 sieve. Conclusions drawn from preliminary tests conducted on proportioning have shown when concrete is produced within these limits a good workable mix can be obtained. The actual percentages depended upon the properties of each mix.

The volumes used were expressed according to

$$\% \text{ Fines} = \frac{\text{Fine Aggregate}}{\text{Total Aggregate}} \times 100$$

where: Fine Aggregate = the volume of the fine aggregate expressed according to the "dry vibrated density" method,

and Total Aggregate = the volume of the total aggregate = the volume of the fine + the volume of the coarse aggregate.

The four levels of the volume of the fine aggregate expressed in terms of percent fines were 55.55, 59.32, 62.50 and 65.22 percent. It should be noted that these percentages are on the high side of the limits of forty to sixty-five percent. One reason for this was that the preliminary tests showed mixes with these high percent fines produced good

workable concrete whereas mixes of lower percent fines produced harsh concrete. Another reason for this arises from the method in which the percent fines is expressed. The volume of the fine aggregate as expressed by the "dry vibrated density" is equal to the sum of the volumes of each size fraction. The "dry vibrated density" was determined for each size fraction and not for each gradation. Therefore, the total volume occupied by each gradation would be somewhat less than that expressed by the sum of the individual size fraction volumes. This is due to the voids produced by the packing of the equal sized particles in the "dry vibrated density" test. These voids would be filled by the smaller size fractions if the test were run on the various gradations.

Cement Factor and Water Cement Ratio

Since bridge decks are more susceptible to the deterioration caused by freezing and thawing, the cement factor was set to comply with the requirements of a conventional bridge deck concrete. The Indiana State Highway Commission Standard Specifications of 1965 specified a cement factor of seven bags of cement per cubic yard of concrete for bridge decks. Therefore, a cement factor of seven bags per cubic yard of concrete was used throughout this experiment. This factor was held constant as closely as possible for all of the mixes.

It was impossible to hold this right on seven bags per cubic yard because each of the 64 mixes was different, each variable affecting the yield in a different way. The average cement factor was 7.25-bags per cubic yard.

The last variable also contained four levels. The four water/cement ratios used were .30, .35, .40 and .45. It should be pointed out that these water/cement ratios were approximate values since it was not possible to determine exactly how much water the lightweight aggregate requires.

Batching, Mixing and Placing

The fine aggregate was separated on a Gilson sieving machine into the various size fractions previously mentioned. These size fractions were re-combined such that the levels of the factors, gradation and volume, were satisfied for each individual mix. Since gradation and volume of fine aggregate were variables, it was of great importance to be able to batch the size fractions with assurance of producing the different level combinations of the variables. The aggregate size fractions were oven-dried and contained no moisture. The fine aggregate was batched by volume on a weight basis using the "dry vibrated densities" of each size

fraction. The weight of each batch was controlled to the nearest gram.

The limestone coarse aggregate, cement and water were batched by weight to the nearest .01 of a pound. The air entraining agent was mixed prior to its use with nine parts of deionized water and stored in a tight glass container. This ten percent mixture was used as required by measuring the proper amount with a small graduated cylinder.

The concrete was mixed in a tub-type mixer of 1-1/2 cubic foot capacity. The limestone coarse aggregate and the fine aggregate were added to the mixer's tub. Then, approximately two-thirds of the water, without the air entraining agent was added. The three components were then mixed for one minute. At the end of this time, the cement was added and mixing continued for one more minute. The other one-third of the required water with the proper amount of air entraining agent was then added and mixed for two minutes at which time the mixing was completed. The air content was then determined along with the mix's yield. If the required amount of air was entrained, the concrete was then placed.

The following specimens were molded from each mix: two 3"x6" cylinders; one 3"x4"x16" beam and one 7"x9"x3" pad.

The concrete was placed in the molds according to ASTM C 192-65, Standard Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory, with the exception of the 7"x9"x3" pads which were rodded fifty times on each of the two layers instead of one per each two square inches of surface. Since the specimen had a large area with a shallow depth, thirty-two penetrations of the rod were insufficient to produce a consolidation equal to that obtained in the 3"x6" cylinders and 3"x4"x16" beams.

Testing

Compressive Strength

The compression tests were conducted according to the ASTM Method of Test for Compressive Strength of Molded Concrete Cylinders, ASTM Designation: C 39-64.

A hydraulic testing machine was used to load the molded 3"x6" concrete cylinders to failure.

Air Content

The air content of each mix was determined by a "Chase" meter. Air contents were checked by the linear transverse method on some of the hardened concrete specimens.

Freeze-Thaw Test

All freeze-thaw testing was conducted according to the ASTM Method of Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water, ASTM Designation: C 291-61T, with the exception of the minimum temperature obtained. The Specifications require that the temperature be lowered to 0 degrees Fahrenheit in three hours or less. This was not possible due to the insulating properties of the lightweight fine aggregates.

The freeze-thaw machine was operated at a capacity of thirty-two 3"x4"x16" concrete beams placed on end. The use of dummy beams was required to maintain the capacity of thirty-two beams. The length of the cycle was four hours. During the freezing phase of the cycle in which the beams were surrounded by air, the temperature of the air was reduced from forty degrees ± three degrees Fahrenheit to 0 degrees ± three degrees Fahrenheit. The freezing phase of the cycle was set at three hours. During the thawing phase the specimens were surrounded by water at a temperature of forty degrees ± three degrees Fahrenheit for a period of fifty minutes. The remaining ten minutes of the cycle were required for draining the thaw water from the freeze-thaw chamber.

The end point for exposure adopted for this test was sixty percent relative dynamic modulus of elasticity or 300 cycles of freezing and thawing, whichever occurred first.

Scaling

The scaling test consisted of freezing concrete pads with approximately one-fourth inch of water on their surfaces then thawing with calcium chloride. The physical dimensions of the pads were 7"x9"x3" with a 1"x3/4" coping around the top surface to permit retention of water.

The freezing phase of the cycle consisted of placing the pads in a walk-in freezer and pouring a measured volume of tap water on their surfaces in order to produce a depth of approximately one-fourth inch. The pads were subjected to the constant temperature of 0 degrees Fahrenheit for a period of four hours at which time the thawing phase of the cycle started. The thawing phase consisted of removing the pads from the freezer and sprinkling a known volume of calcium chloride on their surfaces to produce a concentration of 2.5 percent. The pads were subjected to the laboratory room temperature of approximately seventy-five degrees Fahrenheit for a period of four hours of which approximately ten minutes of this cycle was used to flush the surfaces with clear water to prepare them for the start of another

freeze cycle.

Pictures were taken at different intervals of exposure so that a record of deterioration could be kept. The end point for exposure adopted for this test was 200 cycles of freezing and thawing with a de-icer.

Thermogradient

Thermocouples were embedded in two specimens to determine the rate of freezing, minimum temperature obtained, and the possibility of super-cooling. A thermocouple was embedded in the center of a 3"x4"x16" beam and subjected to cycles of freezing and thawing as were the other specimens according to ASTM C 291-61T, Tentative Specifications for Resistance of Concrete Specimens to Freezing in Air and Thawing in Water. The rate of freezing and the minimum temperature were determined.

Two thermocouples were embedded in a 7"x9"x3" pad, one at the center and one just below the surface. The pad was subjected to cycles of freezing with water on its surface and thawing with calcium chloride as were the other pads. The rate of freezing, minimum temperature and the possibility of super-cooling were determined.

Methods of randomizations were used to obtain mix number 37 as a representative of all the mixes. This mix was

reproduced in exact detail except for the installation of the thermocouples.

Porosity

A Mercury Porosimeter in conjunction with a filling device was used to determine the distribution of the pore sizes.

Representative samples of the No. 4 and No. 8 size fractions of the two lightweight aggregates were tested to determine the pore size distribution and, in most cases, porosity down to the 120 angstrom size. The largest pore size determined was 400 microns.

RESULTS

Compressive Strength

The data obtained from the compressive strength test are presented in Appendix III. The analysis of variance table for the lightweight fine aggregate concrete is presented in Table 6. The method of obtaining the analysis of variance is presented in Appendix I. The calculations involved were not included for this test. For an example of the calculations involved, a complete analysis of one dependent variable, the durability factor, is presented in Appendix IV.

The statistical analysis of the compressive strength showed the levels of the factors aggregate type and water/cement ratio to be significantly different within themselves. The noncoated aggregate concrete produced lower compressive strengths than the coated aggregate concrete. As was expected, the higher the water/cement ratio, the lower the compressive strength.

The average compressive strengths for the coated and noncoated fine aggregate concrete specimens were 7130 and 4740 psi, respectively. It was possible to compare the

TABLE 6

STATISTICAL ANALYSIS OF THE COMPRESSIVE STRENGTH

ANOV Table

Source	DF	MS	MS/SSE*	$F_{0.05;r1,r2}$
A	1	91.49158	19.99**	4.28
B	3	3.42032	--	3.03
C	3	83.46506	18.22**	3.03
D	3	0.93042	--	3.03
AB	2	1.22786	--	3.42
AC	2	.21910	--	3.42
AD	2	.38802	--	3.42
BC	8	2.05326	--	2.37
BD	8	3.62890	--	2.37
CD	8	5.85980	1.28	2.37
Remainder	23	4.58135	1.00	--
	63			

A = Aggregate Type

B = Gradation

C = Water/Cement Ratio

D = Volume of Fines

*SSE = Remainder = 4.58135

**Significant at five percent α level

results of the concrete made with the control sand with the results of eight of the mixes made with the noncoated fine aggregate. The control sand produced concrete with an average compressive strength of 5700 psi and the eight comparable mixes made with the noncoated aggregate produced concrete with an average compressive strength of 4820 psi.

Air Content

The entrained air content was checked for each mix by means of a "Chase" meter. Since air content was not a variable, any mix which did not meet the requirements was disposed of and re-mixed. The limit set for the entrained air content was from two to three percent by volume of the concrete.

The air content was checked on some of the hardened concrete specimens by means of the linear transverse method. It was found that there existed a large amount of entrapped air voids in some of the specimens. The average air content of the nine specimens checked was 3.19 percent, which includes the entrapped air voids. This value was high but, if the entrapped voids were not included, it would be within the two to three percent limit employed.

Freeze-Thaw

The freeze-thaw test was conducted according to ASTM C 291-61T, Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water. The relative dynamic modulus of elasticity was determined at regular intervals and the durability factor was calculated for each beam.

Using the data obtained from the freeze-thaw testing, curves were plotted for each specimen relating relative dynamic moduli of elasticity to cycles of freezing and thawing.

Figures 9, 10 and 11 show typical graphs relating relative dynamic moduli of elasticity to cycles of freezing and thawing for the coated aggregate specimens, the noncoated aggregate specimens, and the control sand specimens, respectively.

A durability factor was determined for each beam according to the ASTM Method of Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water, ASTM Designation: C 291-61T. After the durability factors were determined for all the beams tested they were subjected to statistical analysis to determine the significance of the effects of the variables on the durability of the concrete.

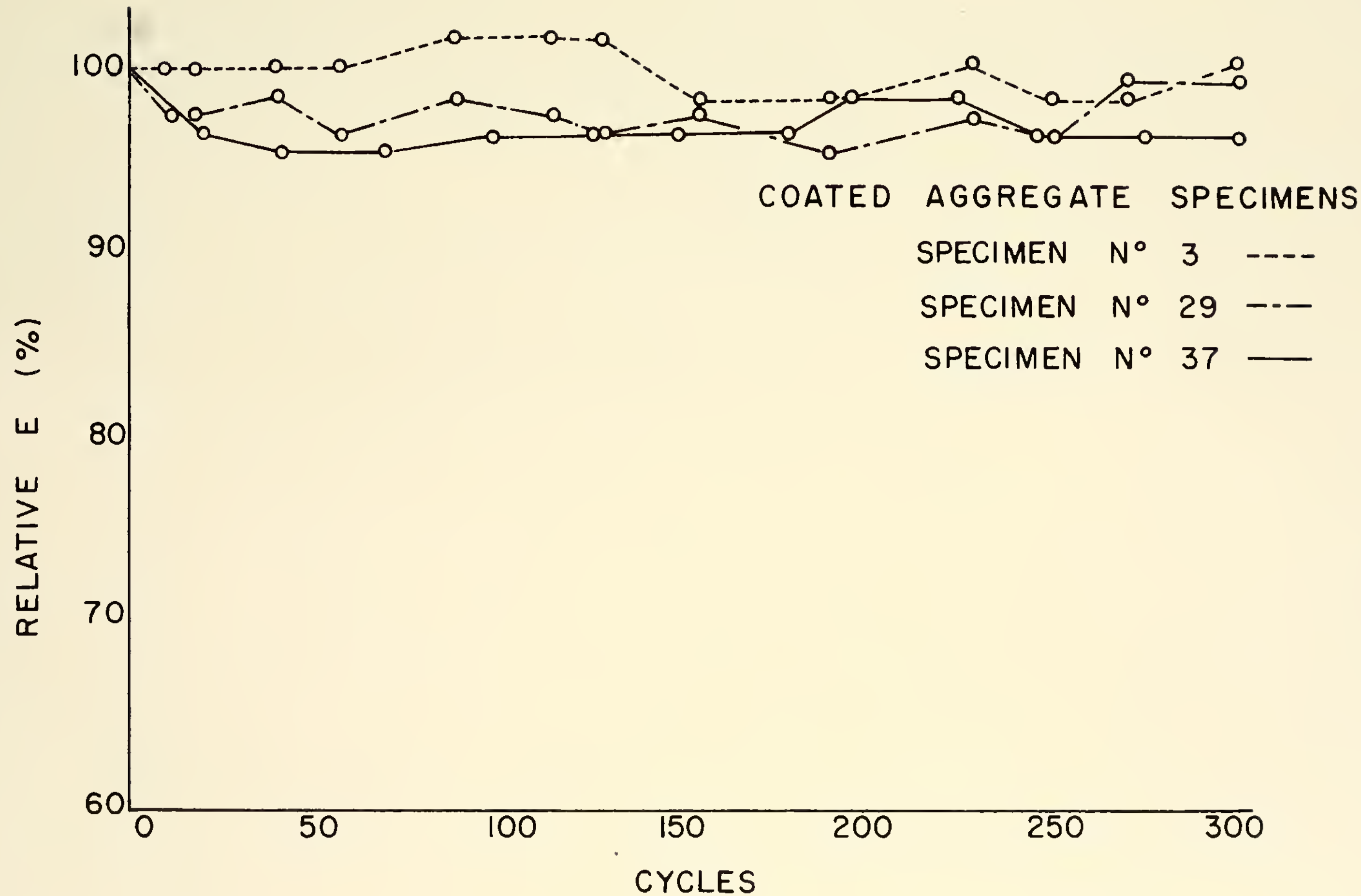


FIGURE 9 RELATIVE E % VS. CYCLES OF FREEZING AND THAWING FOR SPECIMENS 3, 29 AND 37

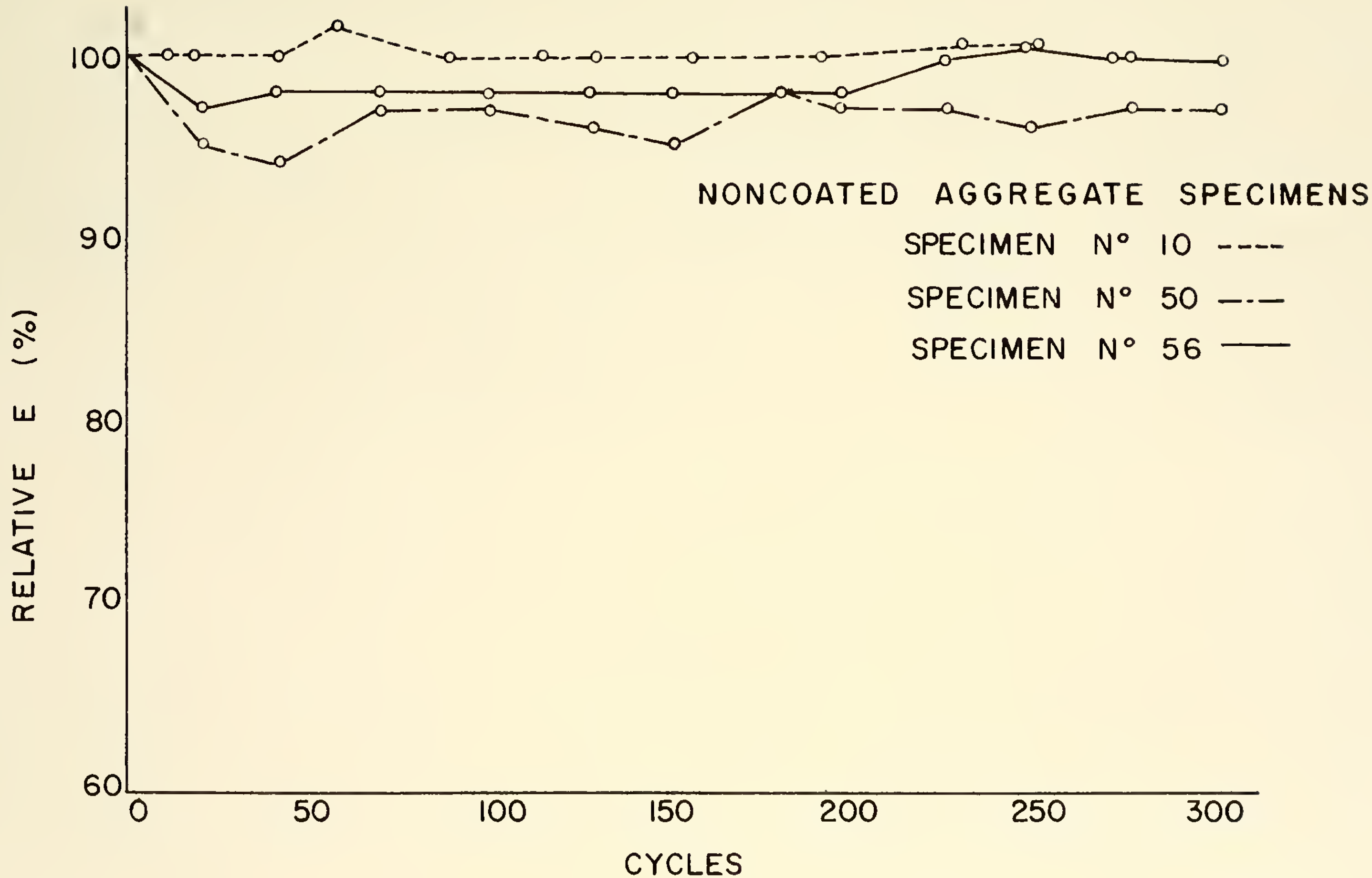


FIGURE 10 RELATIVE E % VS. CYCLES OF FREEZING AND THAWING FOR SPECIMENS 10, 50 AND 56

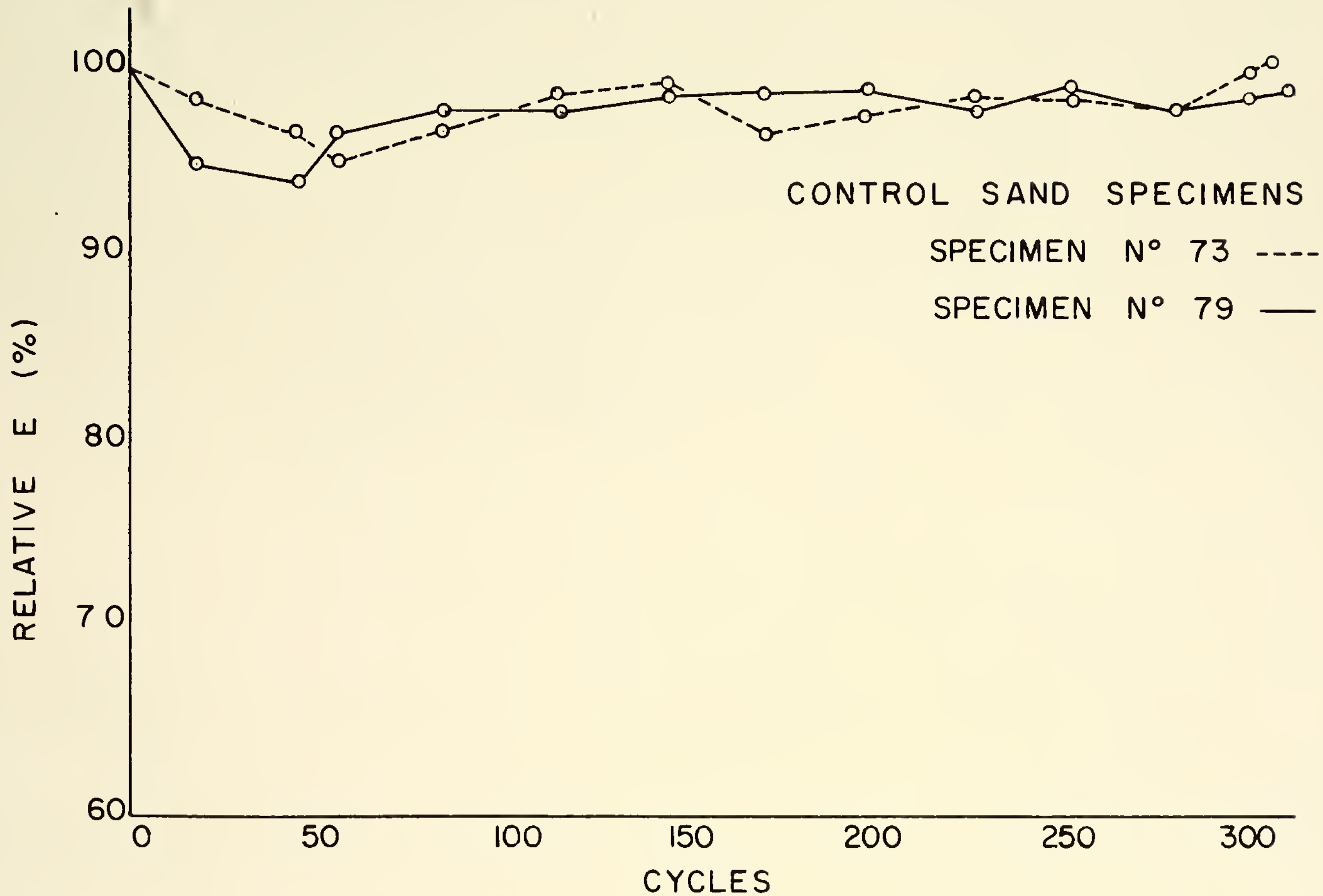


FIGURE II RELATIVE E% VS. CYCLES OF FREEZING AND THAWING FOR SPECIMENS 73 AND 79

The experimental data are presented in Appendix III and the complete analysis of variance is located in Appendix IV. The analysis of variance shows there was no significant difference in the levels of any of the factors tested at the five percent α level.

The average durability factor for the coated aggregate specimens, the noncoated aggregate specimens, and the control sand specimens were 98, 99 and 99 percent, respectively. The durability factors had a range from 91 to 100 percent.

Scaling

The scaling test consisted of freezing water on the surface of each specimen and thawing with a sufficient amount of calcium chloride to produce a 2.5 percent concentration. Pictures were taken of each specimen's surface at various cycle intervals from 00 cycles to 200 cycles. The pictures showed the condition of the specimen surfaces in minute detail. The specimens were rated from pictures as described below.

The statistical consultant recommended that no more than five groups or classes of deterioration be included in the rating system. Table 7 describes the general

description of deterioration for the five rating numbers employed to describe classes of deterioration.

TABLE 7

RATING PROCEDURE

Rating No.	General Description
1	very little noticeable change
2	0 to 33% of the surface gone and/or very shallow pitting
3	0 to 33% of the surface gone and medium pitting or 33 to 67% of the surface gone and very little shallow pitting
4	67 to 100% of the surface gone and/or deep pitting with little to no coarse aggregate exposed
5	67 to 100% of surface gone with deep pitting and little to large percent of coarse aggregate exposed

Figures 12, 13, 14, 15 and 16 present typical pictorial examples of the rating procedure presented in Table 7. It should be pointed out that the pictorial examples shown in these pictures of the rating procedure are not of the quality and large size of the actual pictures used to rate the surface of each specimen. The superior quality and large size (approximately actual size) of the pictures allowed the rating to be performed independent of the actual specimens.

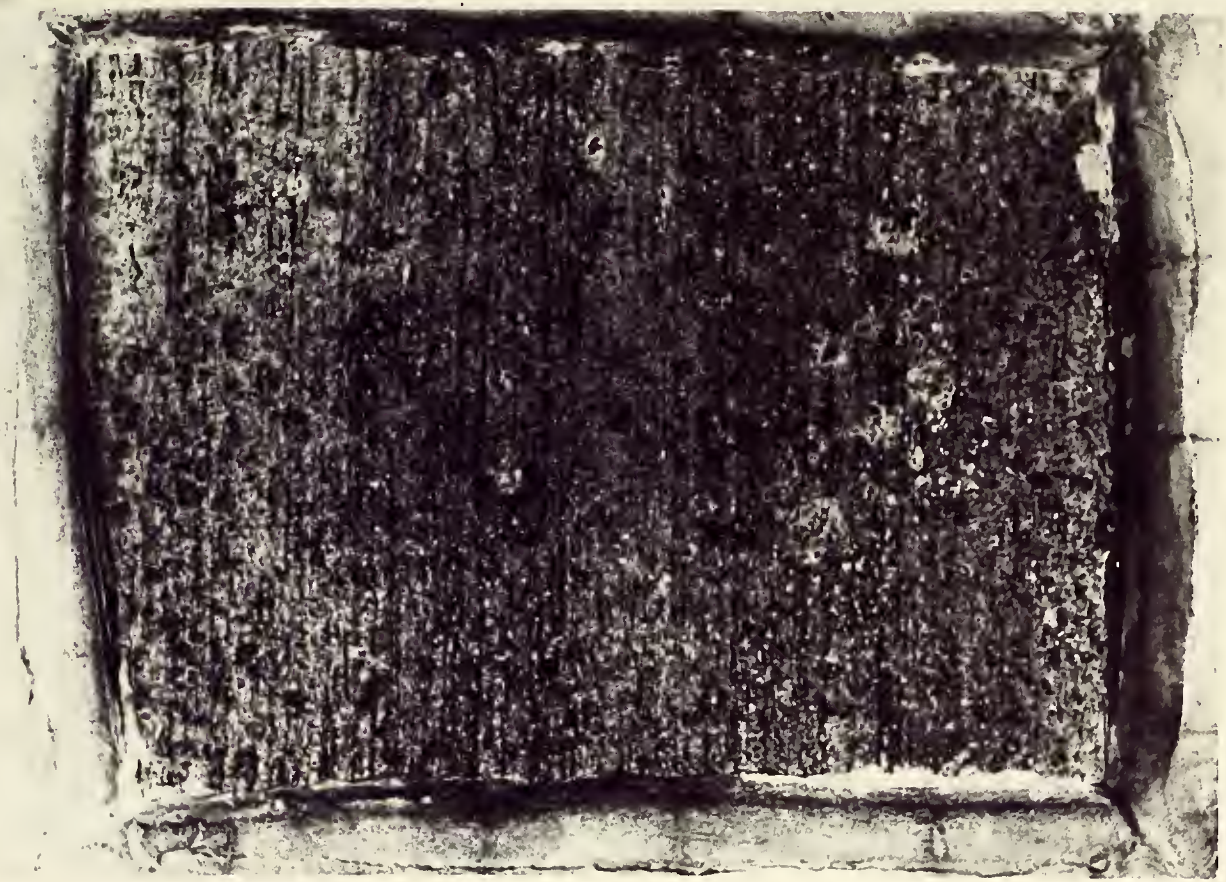


FIGURE 12 PICTORIAL EXAMPLE OF SCALING RATING 1

FIGURE 13 PICTORIAL EXAMPLE OF SCALING RATING 2

FIGURE 14 PICTORIAL EXAMPLE OF SCALING RATING 3

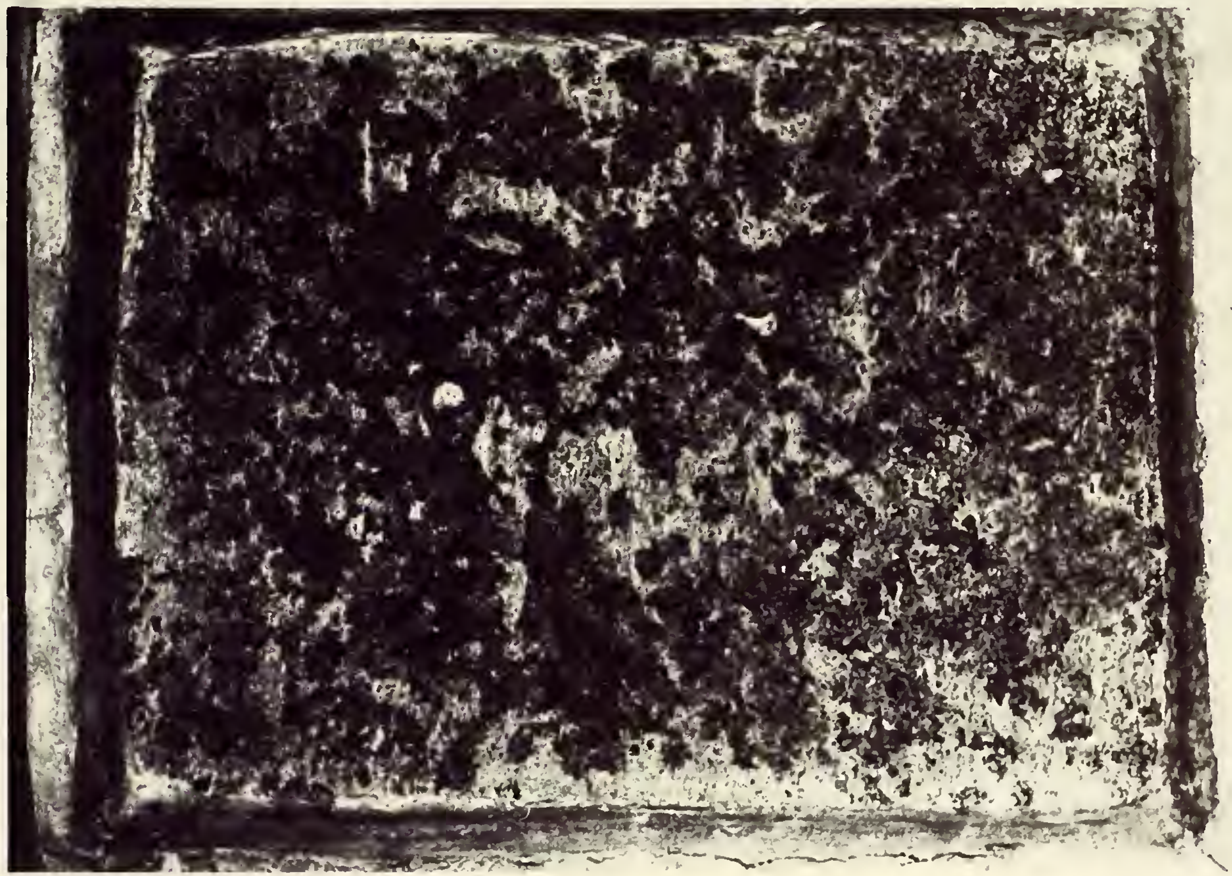


FIGURE 15 PICTORIAL EXAMPLE OF SCALING RATING 4

FIGURE 16 PICTORIAL EXAMPLE OF SCALING RATING 5

It was necessary to utilize the 00 cycle pictures in some cases to determine the extent of deterioration since the grooves were not uniform from specimen to specimen. Non-uniformity of the brooming grooves existed due to the effects of the variable water/cement ratio. A low water/cement ratio produced very shallow brooming grooves whereas the opposite would produce distinct brooming grooves. If the 00 cycle pictures were not used, a low water/cement ratio specimen would appear to have deteriorated more than it really had. Whereas the opposite case would appear in some cases to have deteriorated less than it really had.

Examples of the good, average and bad surface conditions after 200 cycles of freezing and thawing with calcium chloride will be presented for the concrete made with the coated and noncoated aggregates. The examples were rated according to the procedure presented in Table 7. The average is a very good representation of the surface condition of the concrete made from the different aggregate types after 200 cycles. Examples of the surface deterioration of the concretes made with the control sand 79-1G are also included for the good and bad cases. The 00 cycle pictures are included for comparison with the 200 cycle pictures.

Figures 17, 18 and 19 are presented showing the surface deterioration for the specimens made with the noncoated

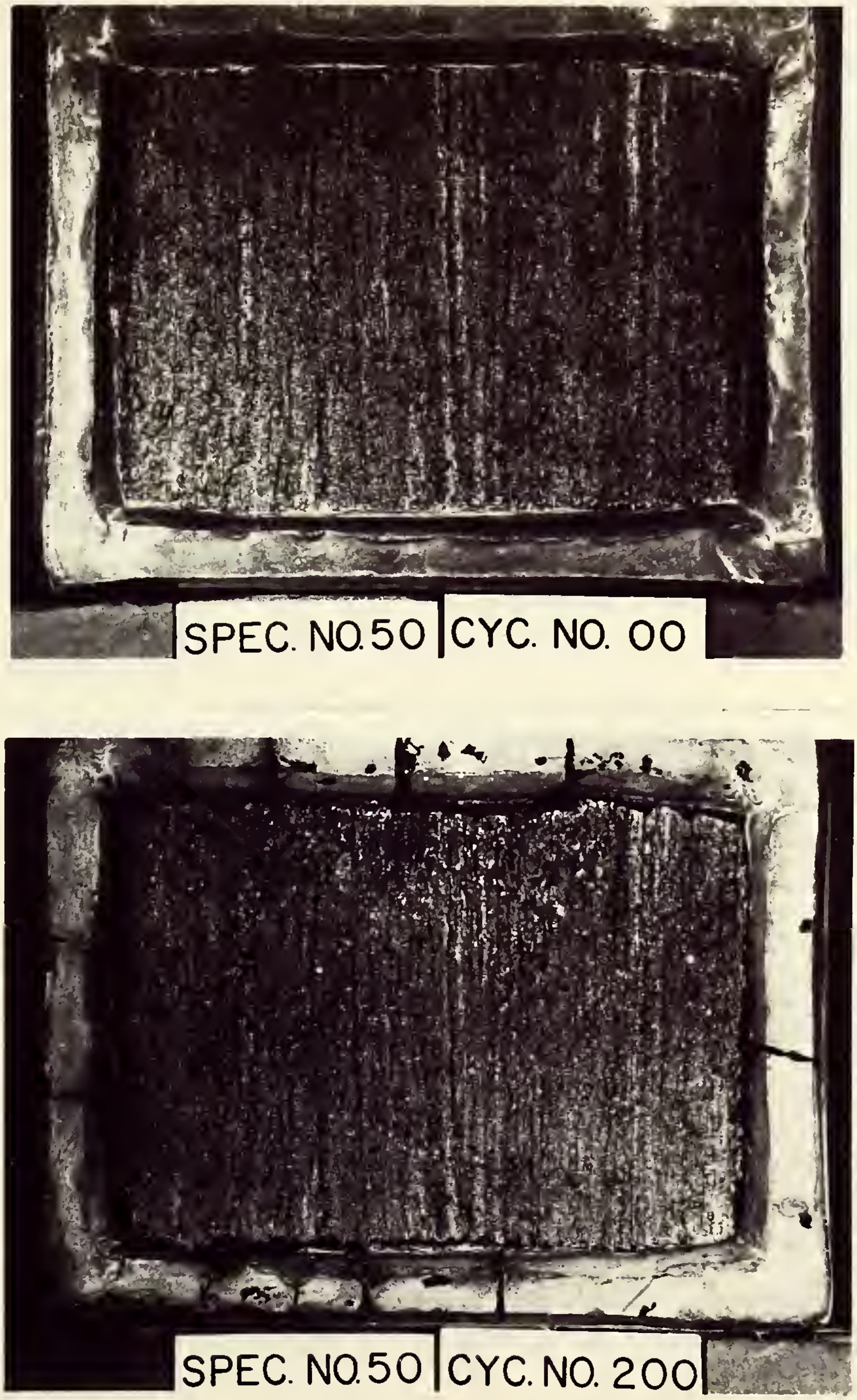


FIGURE 17 SURFACE DETERIORATION OF SPECIMEN 50 AFTER 200 CYCLES OF FREEZING AND THAWING WITH A DE-ICER

FIGURE 18 SURFACE DETERIORATION OF SPECIMEN 10 AFTER 200 CYCLES OF FREEZING AND THAWING WITH A DE-ICER

FIGURE 19 SURFACE DETERIORATION OF SPECIMEN 56 AFTER 200 CYCLES OF FREEZING AND THAWING WITH A DE-ICER

aggregates after 200 cycles of freezing and thawing with calcium chloride for specimens 50, 10 and 56, respectively. These specimens were given ratings of 1, 3 and 4, respectively. Specimen 10 represents the average of the 32 non-coated aggregate specimens. Specimens 37, 29 and 3, representing the coated aggregate concretes, are presented in Figure 20, 21 and 22, respectively. Specimen 37, representing one of the best, was given a rating of 3 and specimen 3 being one of the worst was given a rating of 5. The average of the 32 coated aggregate specimens is represented by specimen 29 which was given a rating of 4. Figures 23 and 24 represent the surface deterioration for the control sand concrete after 200 cycles of freezing and thawing with calcium chloride for specimens 73 and 79, respectively. Specimen 73 was given a rating of 4 and specimen 79 a rating of 1.

The pictures were rated by three observers; the first observer being the only one to use the rating procedure in Table 7. Rather than bias the results of the other two observers, the rating procedure was not given. The only information given was that there should be no more than five groups or classes of ratings.

The results of the three observers were analyzed and were found to give similar results. The statistical analysis

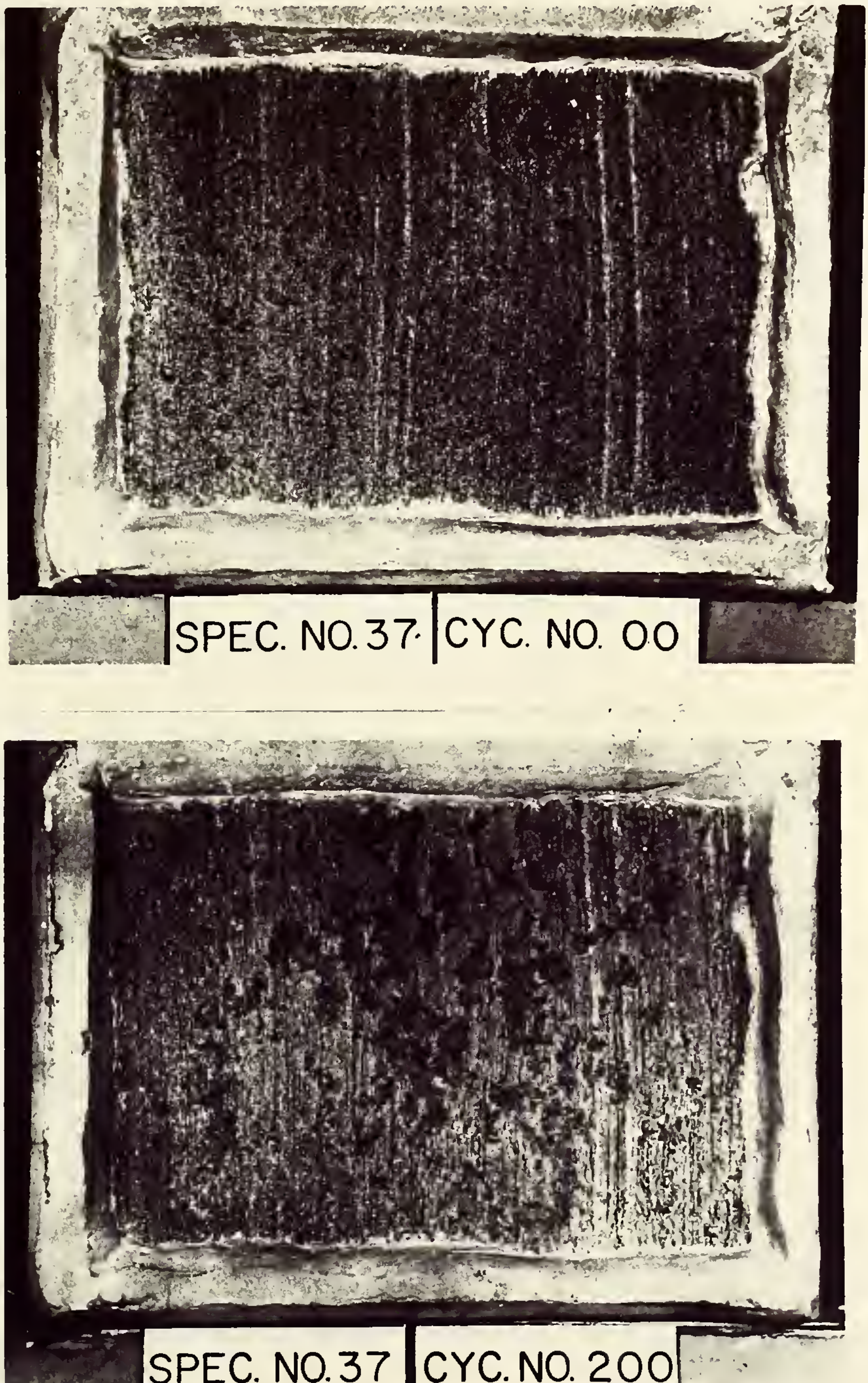


FIGURE 20 SURFACE DETERIORATION OF SPECIMEN 37 AFTER 200 CYCLES OF FREEZING AND THAWING WITH A DE-ICER

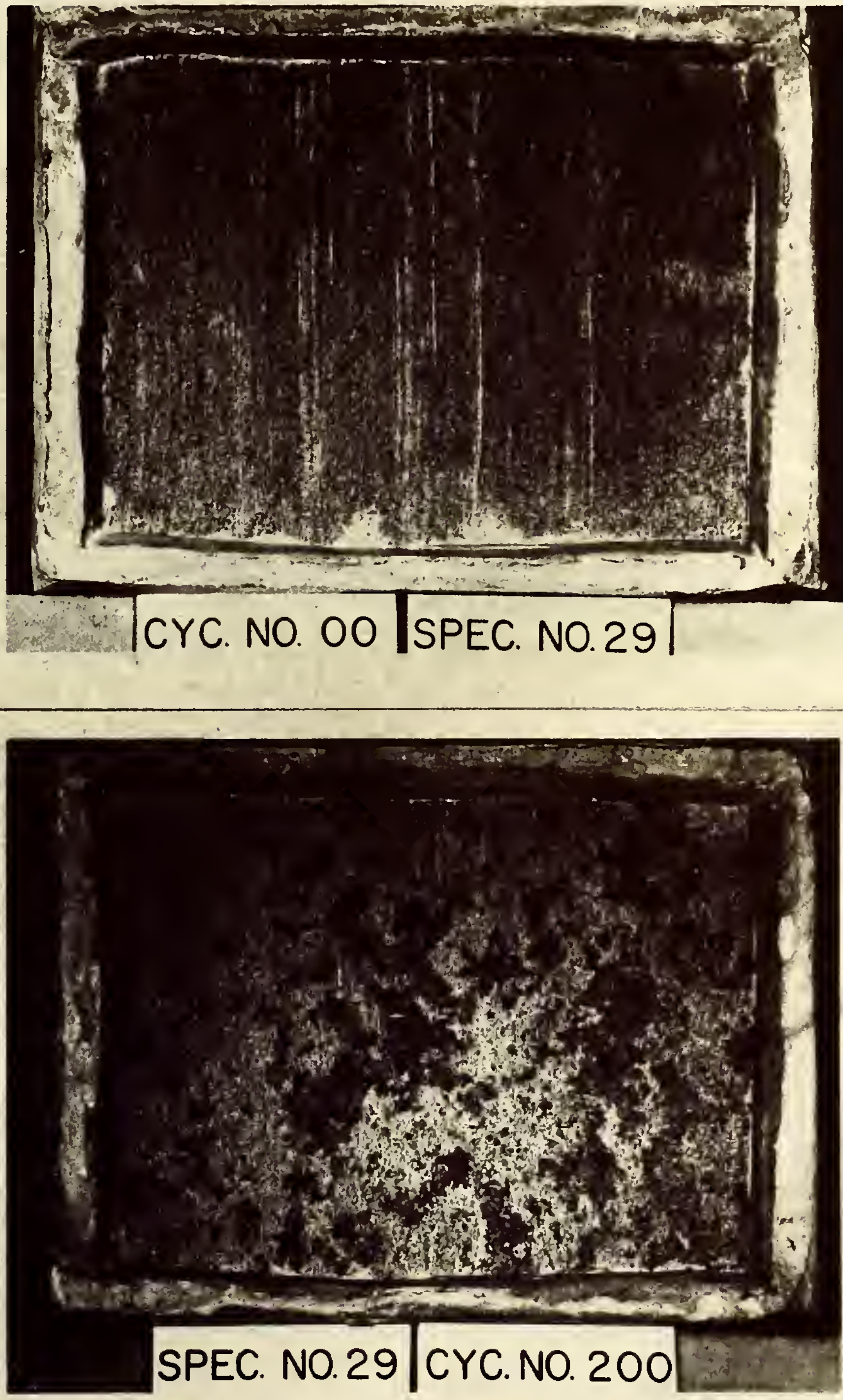


FIGURE 21 SURFACE DETERIORATION OF SPECIMEN 29 AFTER 200 CYCLES OF FREEZING AND THAWING WITH A DE-ICER

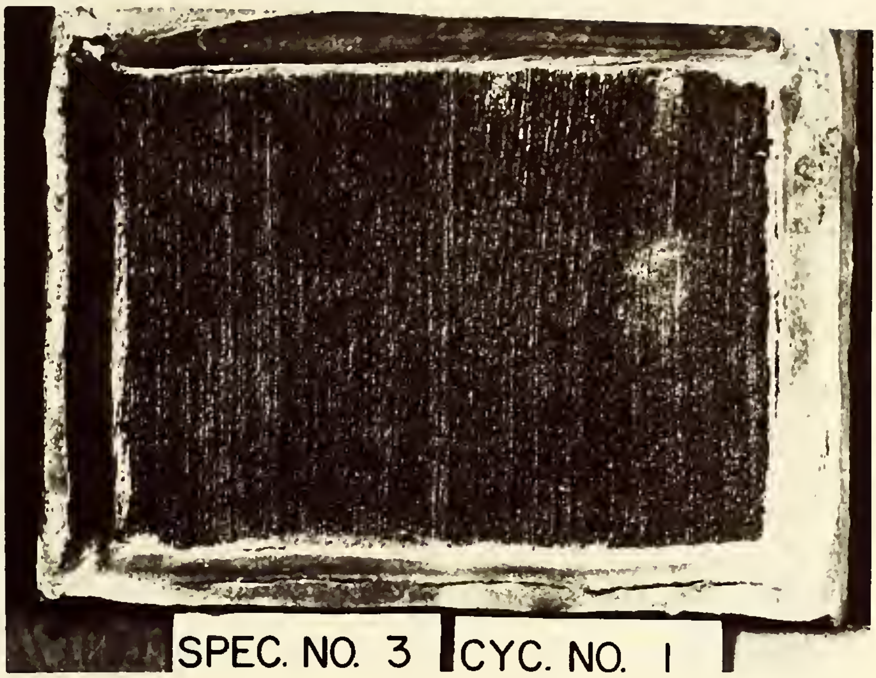


FIGURE 22 SURFACE DETERIORATION OF SPECIMEN 3 AFTER 200 CYCLES OF FREEZING AND THAWING WITH A DE-ICER

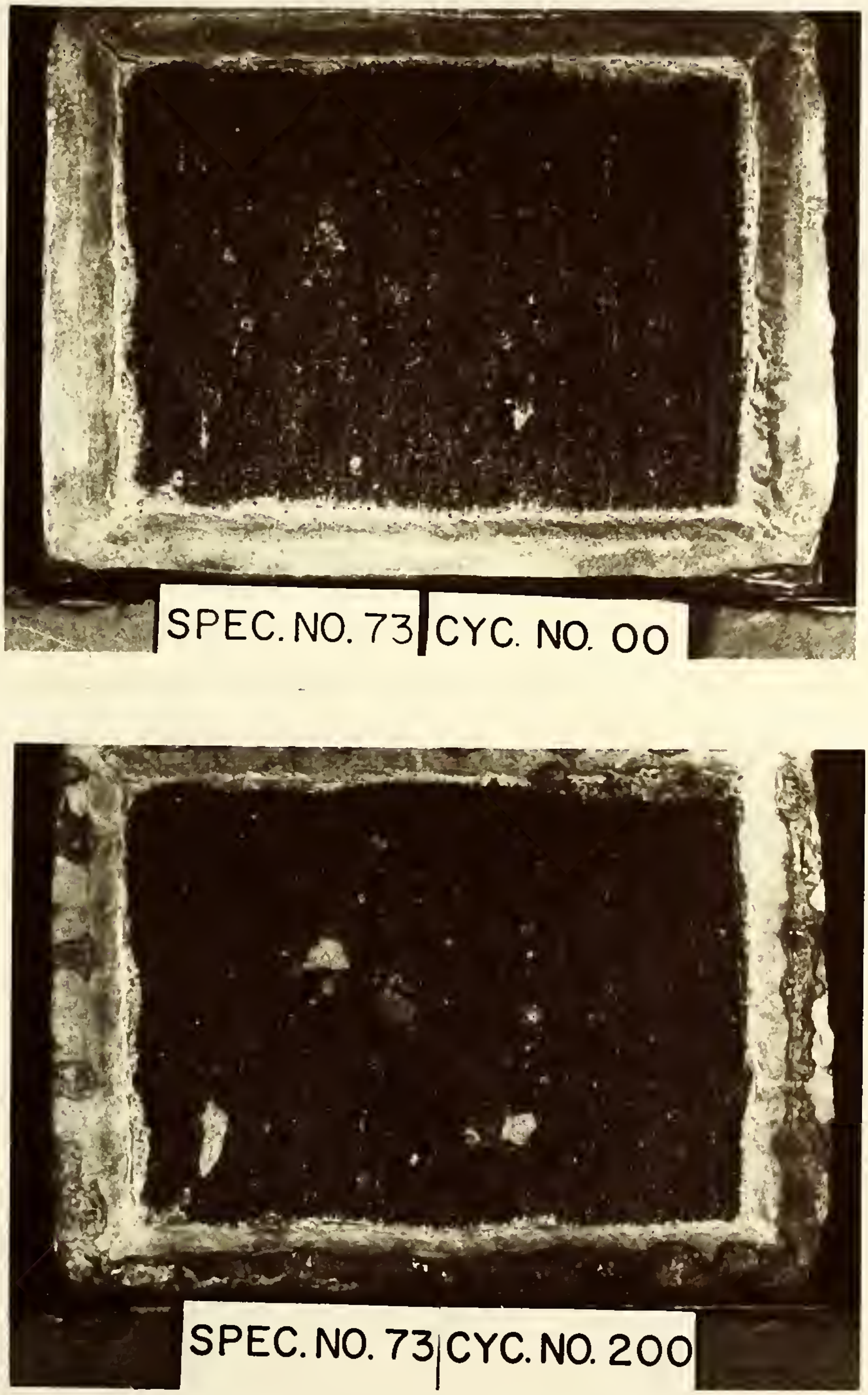


FIGURE 23 SURFACE DETERIORATION OF SPECIMEN 73 AFTER 200 CYCLES OF FREEZING AND THAWING WITH A DE-ICER

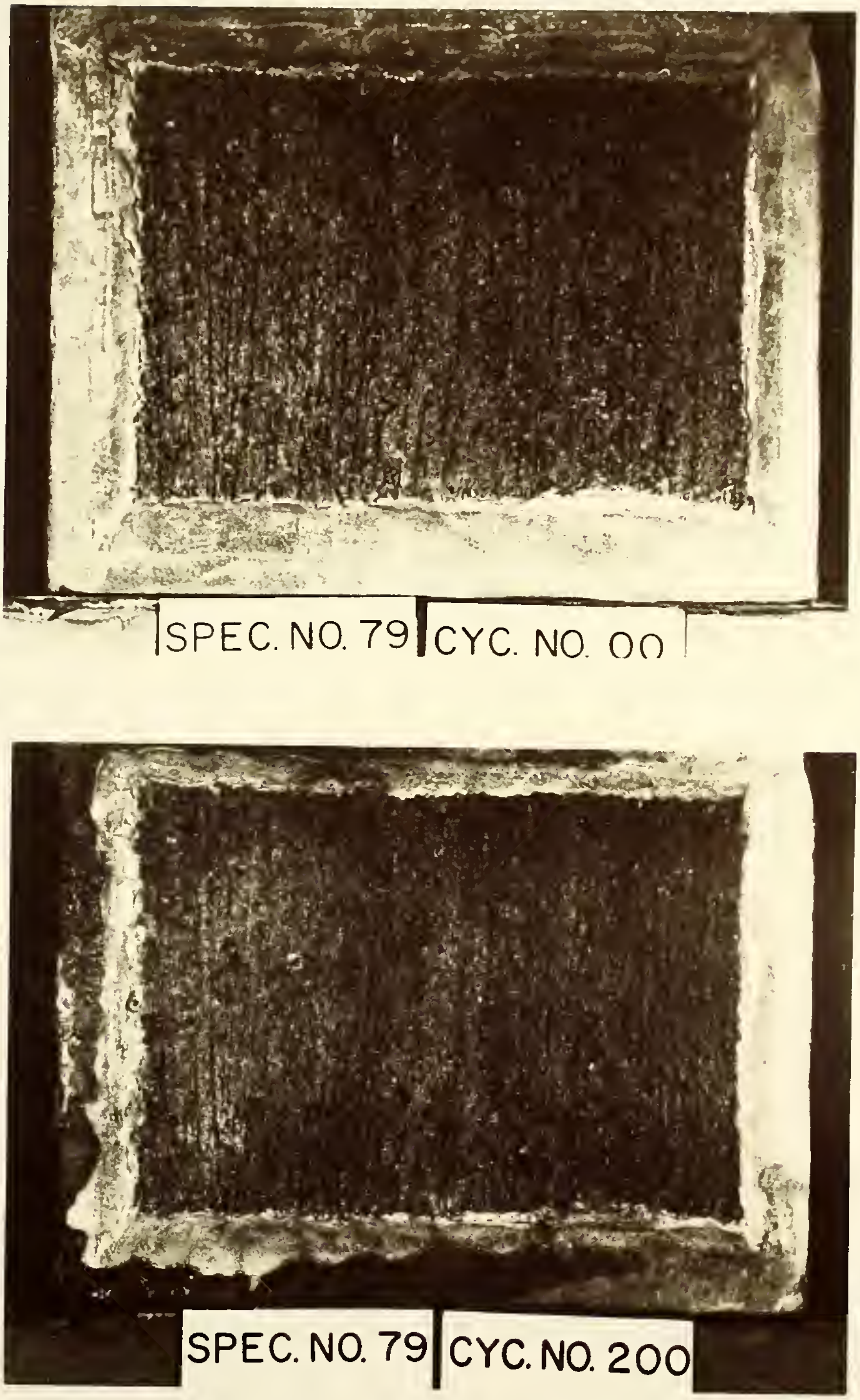


FIGURE 24 SURFACE DETERIORATION OF SPECIMEN 79 AFTER 200 CYCLES OF FREEZING AND THAWING WITH A DE-ICER

of variance for the first observer is presented in Table 8. The other two showing similar results are not included. It is concluded from the analysis of variance of the scaling rating that there were no significant differences in any of the levels of the factors tested at the five percent α level.

A bar graph showing the frequency of scaling rating versus the scaling rating for the coated and noncoated aggregate concrete is presented in Figure 25. The average rating given to the coated aggregate specimens and the noncoated aggregate specimens was 4.0 and 2.8, respectively. The best rating given to any specimen of the coated aggregate concrete was 3 and for the noncoated aggregate concrete a rating of 1. The worst rating given to the coated aggregate specimens was 5 and for the noncoated aggregate specimens a rating of 3. The average specimen of the noncoated aggregate concrete was given a better rating than the best rating given to any of the coated aggregate specimens.

The control sand produced concrete specimens which were given an average rating of 1.9. The equivalent noncoated aggregate specimens were given an average rating of 3.3. The range of the ratings given in both cases was 1 to 4.

TABLE 8

ANOV TABLE FOR OBSERVER NO. 1

Source	DF	MS	MS/SSE*	$F_{0.05;\gamma_1,\gamma_2}$
A	1	21.39062	1.17	4.28
B	3	1.42186	--	3.03
C	3	3.27343	--	3.03
D	3	2.54486	--	3.03
AB	2	5.78124	--	3.42
AC	2	1.90624	--	3.42
AD	2	0.03124	--	3.42
BC	8	3.62496	--	2.37
BD	8	1.99996	--	2.37
CD	8	4.12496	--	2.37
Remainder	23	18.23658	1.00	--
	63			

A = Aggregate Type

B = Gradation

C = Water/Cement Ratio

D = Volume of Fines

*SSE = Remainder Sum of Squares = 18.23658

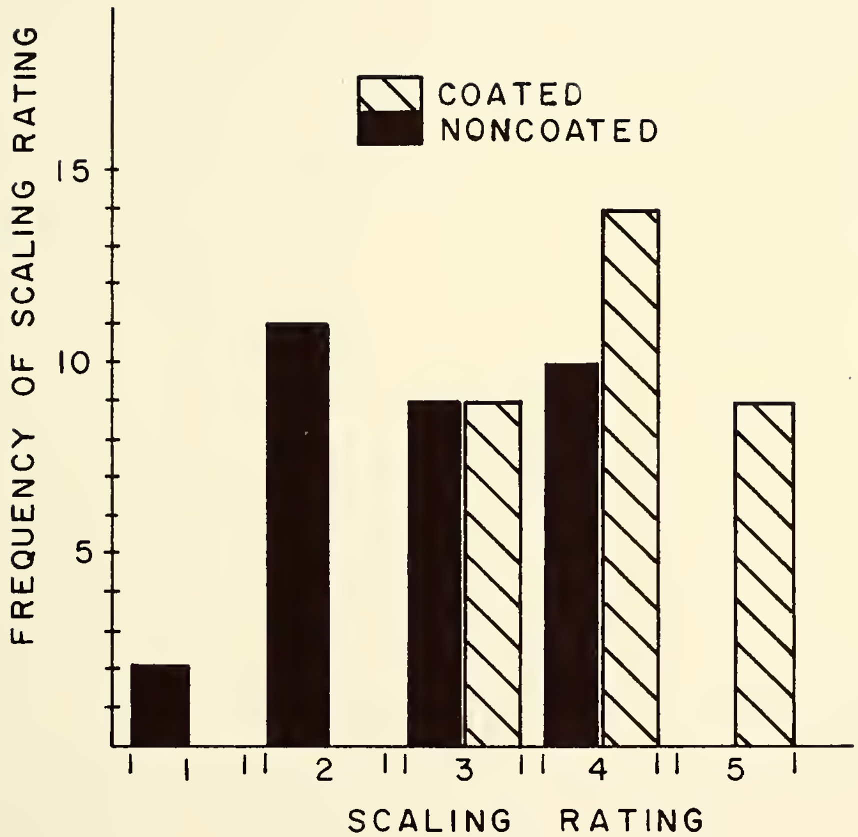


FIGURE 25 FREQUENCY OF SCALING RATING VS. SCALING RATING FOR THE COATED AND NONCOATED AGGREGATE CONCRETES

Thermogradient

Figure 26 shows the temperature fluctuation for the freeze-thaw test. The results are within the specifications of ASTM C 291-61T except for the minimum temperature obtained. The insulating properties of the lightweight fine aggregate prevented the specimen from reaching 0 degrees Fahrenheit. Figure 27 shows the temperature fluctuation for the scaling test. Figure 28 shows the relation between temperature and time for that period of the cycle just before and after thawing was induced.

Porosity

A mercury porosimeter in conjunction with a filling device was used to determine the distribution of the pores and the total porosity. Representative samples of the No. 4 and No. 8 size fractions of the two lightweight aggregates were tested. Figures 29 and 30 show the relation between intrusion of mercury per gram and minimum pore size intruded for the coated and noncoated aggregates, respectively. These tests were run down to the 120 angstrom pore size except the coated aggregate No. 8 size fraction which went down to the 0.4 micron size. A dashed line represents what the continuation of the curve might have looked like if the test had been completed.

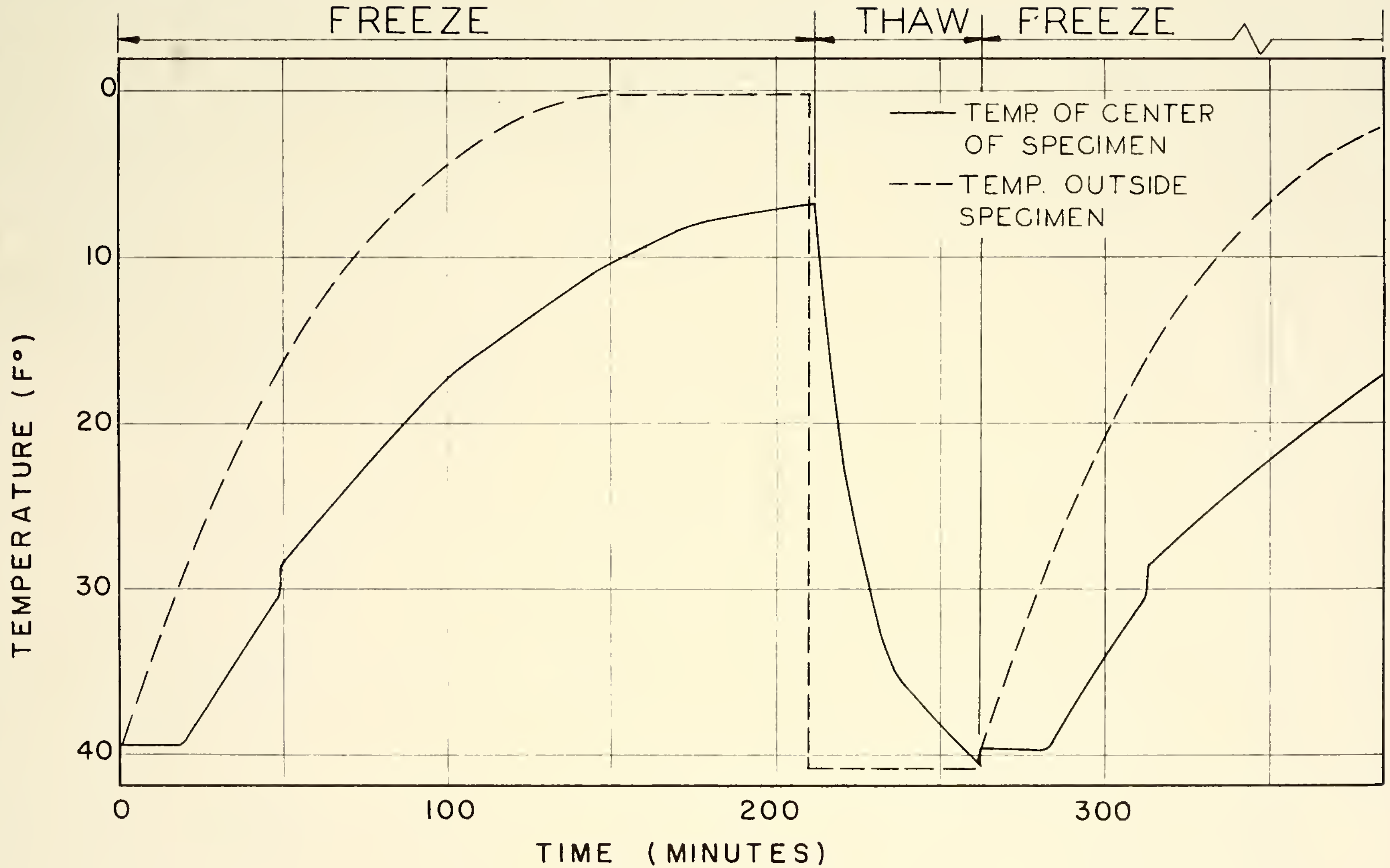


FIGURE 26 TEMPERATURE (°F) VS. TIME IN MINUTES FOR A FREEZE-THAW CYCLE OF 3x4x16" BEAMS

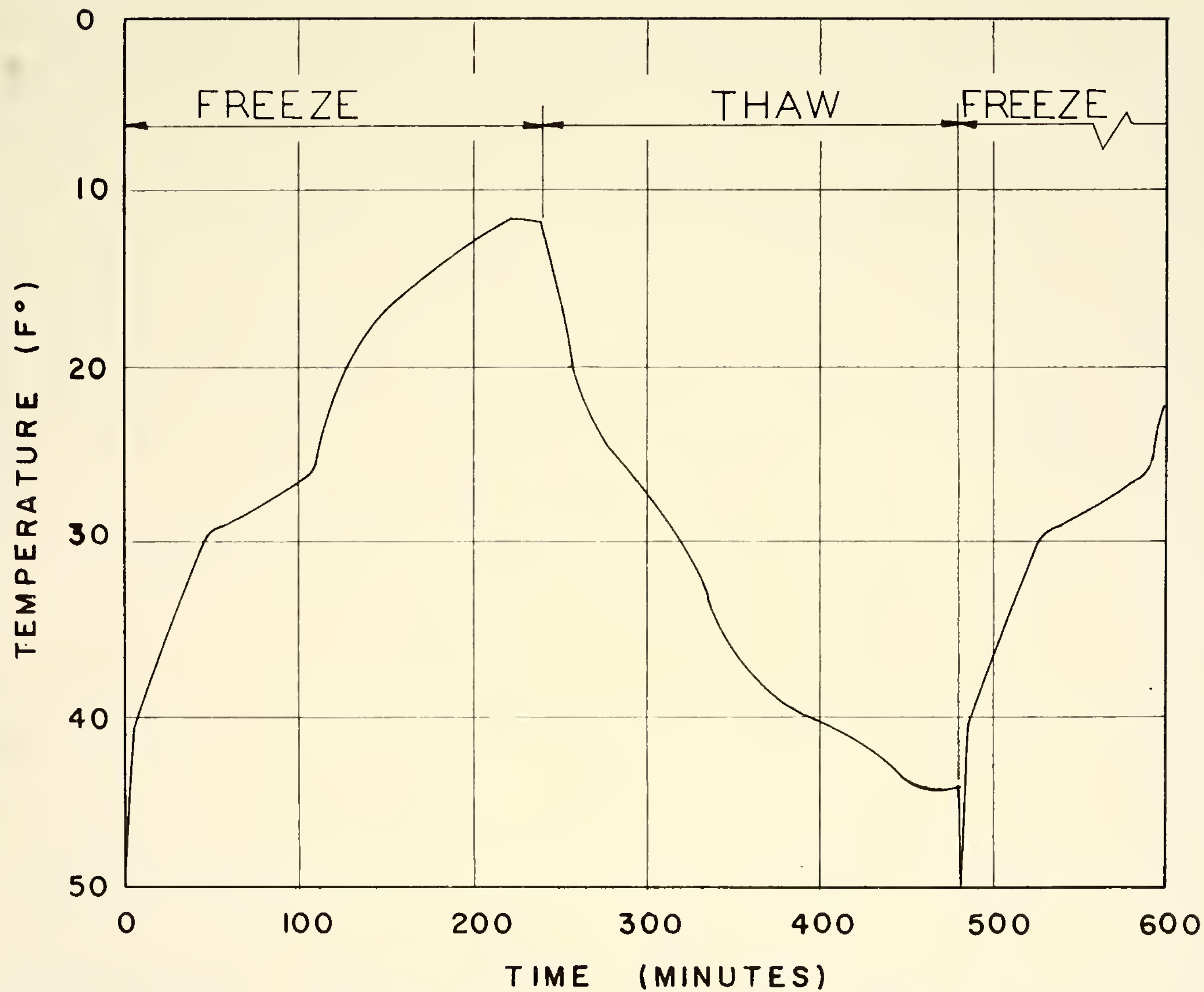


FIGURE 27 TEMPERATURE (°F) VS. TIME IN MINUTES FOR A FREEZE-THAW CYCLE OF 7x9x 3" PADS

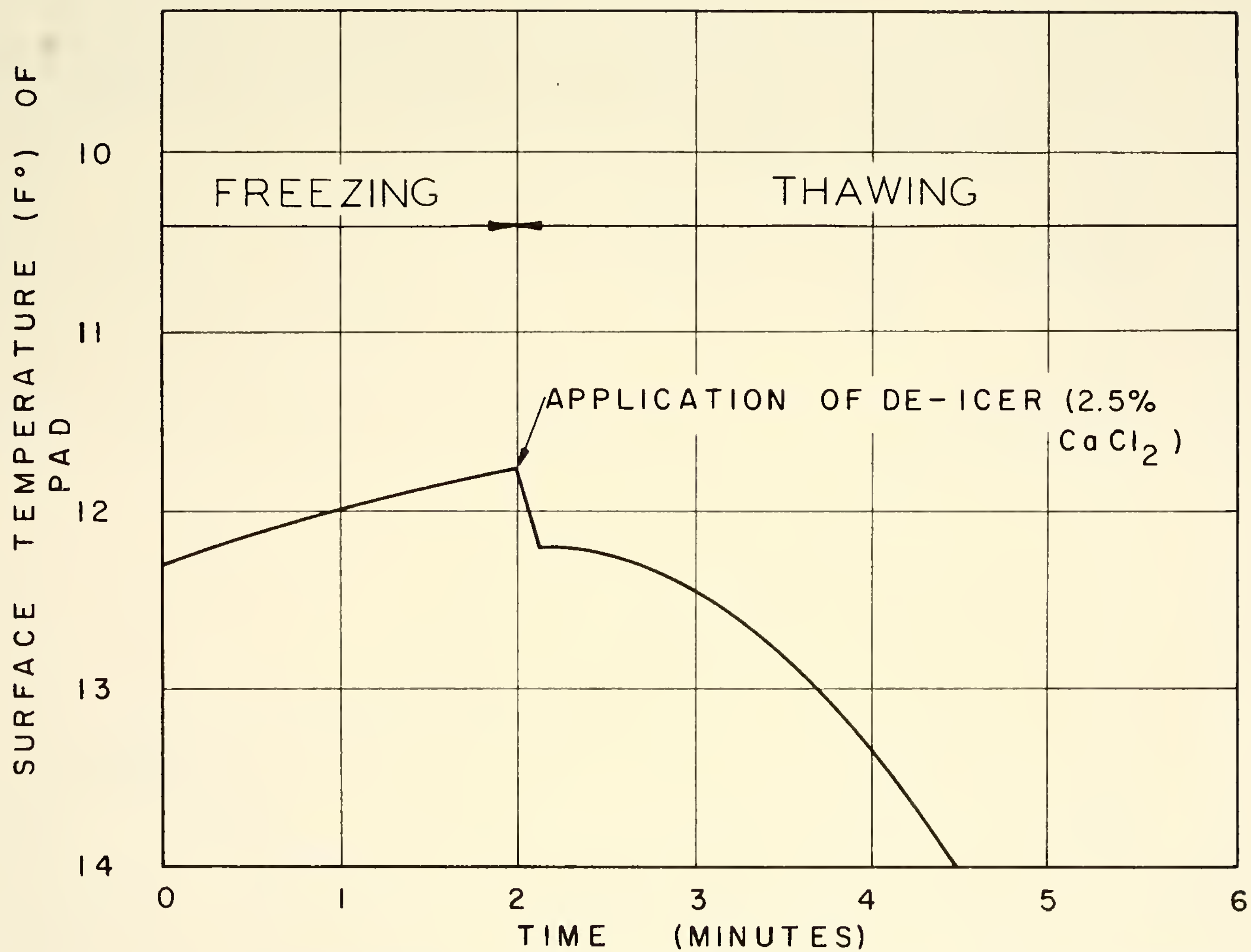


FIGURE 28 SURFACE TEMPERATURE (°F) OF PAD VS. TIME (MINUTES)

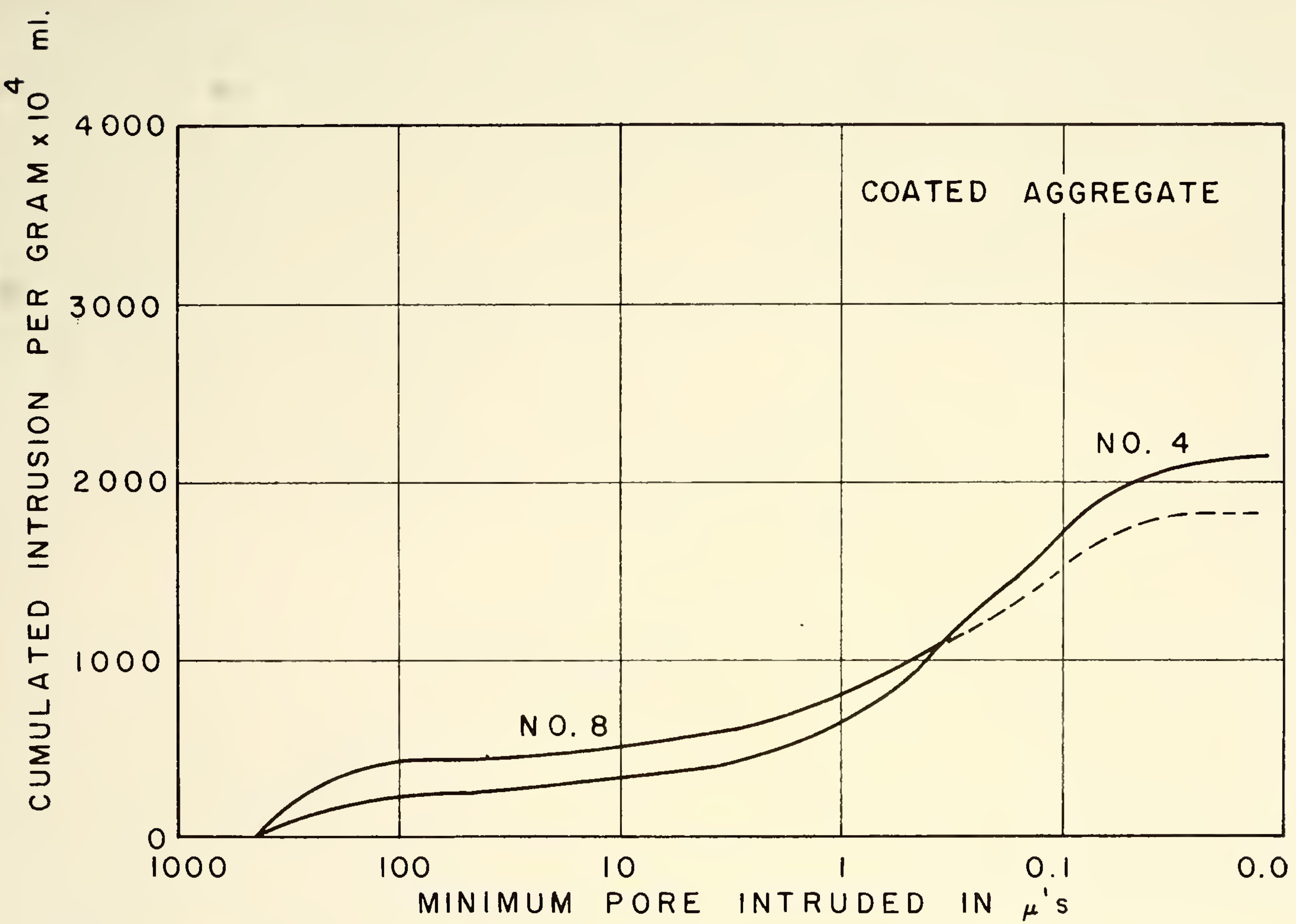


FIGURE 29 CUMULATED INTRUSION PER GRAM x 10^4 ml. VS. MINIMUM PORE INTRUDED IN μ's FOR THE COATED AGGREGATE

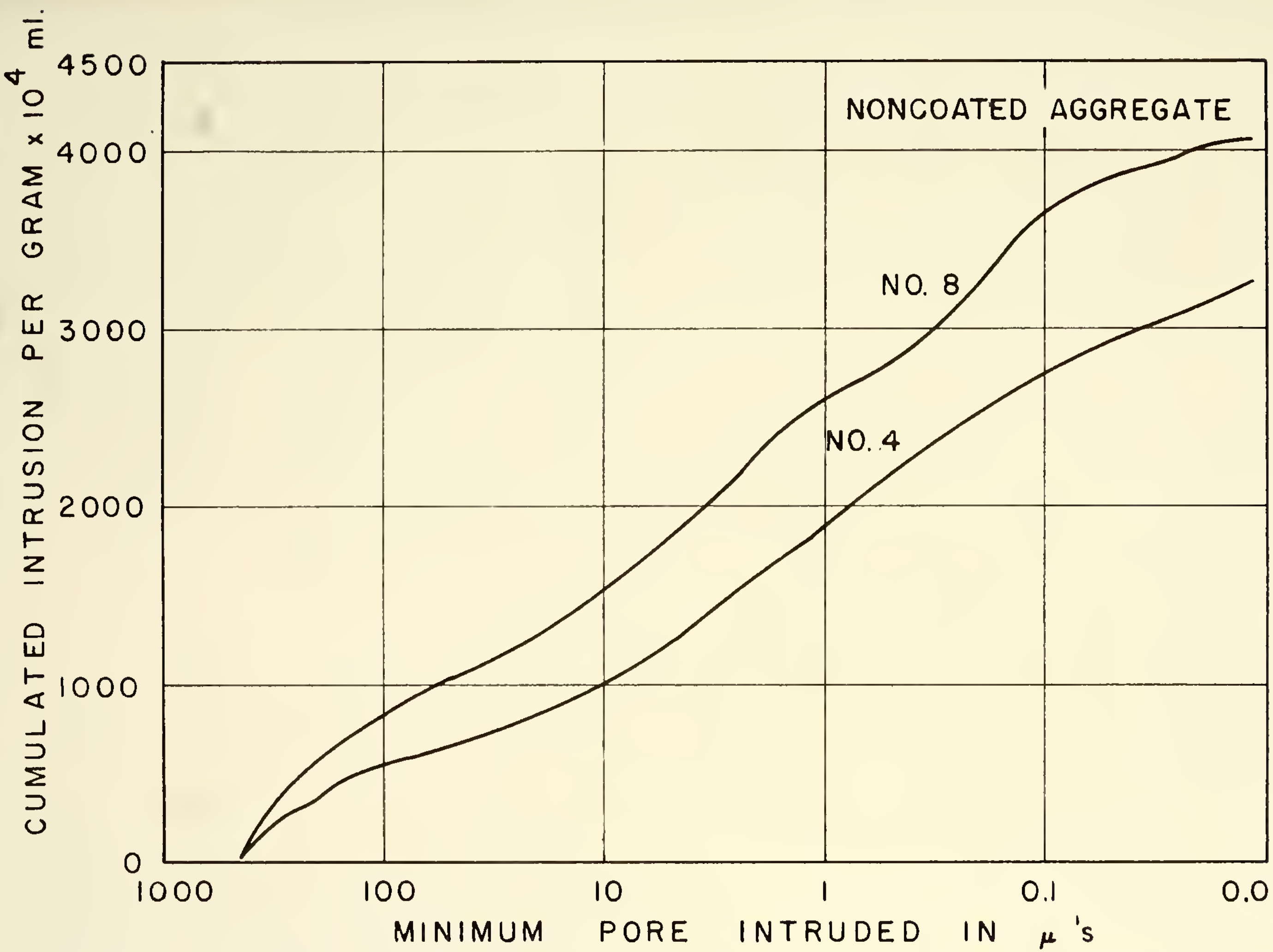


FIGURE 30 CUMULATED INTRUSION PER GRAM x 10^4 ml. VS. MINIMUM PORE INTRUDED IN μ's FOR THE NONCOATED AGGREGATE

DISCUSSION OF RESULTS

Compressive Strength

The statistical analysis of variance showed the variables aggregate and water/cement ratio were significant at the five percent α level. The concrete made with the coated aggregates produced higher compressive strengths than the noncoated aggregates. As was expected, the higher the water/cement ratio the lower the compressive strength for both aggregate levels.

Freeze-Thaw

The statistical analysis of variance of the durability factors showed no significant differences in the levels of any of the factors tested. The average durability factor of the specimens made from the coated aggregate, the noncoated aggregate and the control sand were 98, 99 and 99 percent, respectively.

The durability factor provides an index of the relative freeze-thaw resistance but does not give a good indication of the scaling resistance of concrete. This test is based on a massive or deep seated failure and therefore does not give any indication of the scaling resistance of a concrete.

An example of a concrete capable of withstanding the deteriorating influences of the freeze-thaw test but performing poorly under the scaling test may be seen in Figures 9 and 22. Figure 9 shows the graph of relative E versus cycles of freezing and thawing for specimen No. 3. This specimen was given a durability factor of 100 percent after 300 cycles of freezing and thawing and was considered to be very durable. Figure 22 shows the condition of the surface of specimen (pad) No. 3 after 200 cycles of freezing and thawing with calcium chloride. A rating of 5 was given to this specimen. Therefore, specimen No. 3 shows one case in which the freeze-thaw test did not give an indication to the scaling resistance. In fact all the specimens produced high durability factors but only a few withstood the deteriorating effects of the scaling test. On the basis of the freeze-thaw test any of the specimens tested would produce a good durable concrete.

Scaling

The visual results of the scaling test are not supported by the statistical analysis. A significant difference could not be shown statistically between the coated and noncoated aggregates but there is little question as to whether there really was a difference between the two aggregates. The reason for this is not known but the test employed was of a very conservative nature.

It was possible to test in the analysis of variance whether the assumption of negligible three factor and higher interactions was true or not by testing against the error sum of squares. When this assumption was not found to be true, the remainder sum of squares provided a conservative test for testing the main effects and the effects of two factor interactions. Had it not been significant the error sum of squares would have been used for the testing. The fact that the remainder sum of squares provided a conservative test cannot be over emphasized.

Comparison of the surface deterioration of the specimens shown in the pictures in Figures 17 through 22 is of interest. These pictures are typical for the individual group they represent.

Specimen No. 50, shown in Figure 17, was made with the noncoated aggregate and was given a rating of 1. This represents one of the best specimens made with the noncoated aggregate. Figure 20 shows a concrete made with the coated aggregate and was given a rating of 3. This represents one of the best specimens made with the coated aggregate. The coated aggregate concrete surface developed medium depth pitting. The noncoated aggregate definitely produced the best surface and showed very little change after the 200th cycle of testing.

Specimen No. 10 represents the average rating given to all the specimens made with the noncoated aggregate and was given a rating of 3. Figure 21 represents the average rating given to all the specimens made with the coated aggregate. This specimen was given a rating of 4. Specimen No. 10 developed shallow scaling and Specimen No. 29 not only developed shallow scaling but also deep pitting. The noncoated aggregate produced the most durable surface.

Figure 19 shows one of the worst specimens made from the noncoated aggregate. This was given a rating of 4. One of the worst coated aggregate specimens presented in Figure 22 was given a rating of 5. Specimen No. 56 shows that about eighty percent of its surface has scaled off but only

to a shallow depth. Specimen No. 3 has 100 percent of its surface gone with the scaling deep enough to expose some of the coarse aggregate. The noncoated aggregate definitely produced the better surface for the worst possible case of each aggregate type.

Figures 23 and 24 are representatives of good and bad concrete surfaces made from the control sand. Specimen No. 79, like Specimen No. 50 in Figure 17, showed very little change and was given a rating of 1. Specimen No. 73 was given a rating of 4 because of the exposed coarse aggregate. This specimen was rated the same as the worst case of the noncoated aggregate specimens. Although it is not illustrated by pictures, the average rating given to the specimens made with control sand was 1.9 which compares with an average rating of 3.3 for the equivalent noncoated aggregate specimens. Thus, the average noncoated aggregate specimen was not as good as the average control sand specimen.

A bar graph showing the frequency of scaling rating versus the scaling rating for the coated and noncoated aggregate concrete was presented in Figure 25. The average rating given to the coated aggregate specimens and the noncoated aggregate specimens was 4.0 and 2.8, respectively. The best rating given to any specimen of the coated and

noncoated aggregate concretes was 3 and 1, respectively. The worst rating given to the coated aggregate specimens was 5 and for the noncoated aggregate specimens a rating of 3. The average rating given to the specimens made from the noncoated aggregate was appreciably better than the best rating given to any of the coated aggregate specimens.

The control sand produced concrete specimens which were given an average rating of 1.9. The equivalent noncoated aggregate specimens were given an average rating of 3.3. The range of the ratings given in both cases was 1 to 4.

It was impossible to predict the relationship among the levels of the variables tested were because statistically none of the levels were different.

It was concluded from these results that the noncoated aggregate produced concrete superior to the coated aggregate, and the control sand produced concrete better than the average made from the noncoated aggregate.

Porosity

Many mercury porosimeter tests were run but only a few were completed down to the 120 angstrom pore size. Some peculiar characteristics of the lightweight aggregates caused the column of mercury in the penetrometer to separate

when subjected to the high pressures of the mercury porosimeter. When the column of mercury separated the test had to be terminated since the amount of mercury intruded with increased pressures could not be determined. The actual cause of separation was unknown.

The graphs show total cumulated intrusions per gram of 3250 and 4050 x 10^{-4} ml. for the No. 4 and No. 8 size fraction of the noncoated aggregates. The graph of the No. 4 size fraction of the coated aggregate shows a total cumulated intrusion per gram of 2120 x 10^{-4} ml.

It has been reported that the pores smaller than 5 microns are important predictors of a coarse aggregate's freeze-thaw durability (19). The noncoated aggregate No. 4 and No. 8 size fractions had 63 and 56 percent of their total pores less than 5 microns, respectively. The No. 4 size fraction of the coated aggregate had 82 percent of its total pores less than 5 microns. It should be pointed out that these figures are based on the pores between 450 microns and 120 angstrom.

Comparing the noncoated aggregate's most porous size fraction with that of the coated aggregate's implied that the coated aggregate had a porosity approximately half that of the noncoated aggregate. It was also found that the coated aggregate had approximately one and one-half the percentage

of pores smaller than 5 microns, on a total pore basis, than did the noncoated aggregate.

If this experiment had shown differences in the level combination of the variables, it would have been feasible to predict properties of the "solid bubble". It was possible to predict what range of size fractions should produce protection of the paste during freezing using the data obtained from the porosity test. This range of size fractions could not be confirmed from the data of this experiment.

Influence of Experimental Factors

This experiment was designed to determine the effect of gradation, water/cement ratio and volume of fines on the freeze-thaw and scaling resistance of concrete made with two lightweight fine aggregates with conventional coarse aggregate. These variables were chosen such that the results would predict properties of the "solid bubble". Gradation was chosen as a variable to determine the effect of the individual size fractions and volume of the fines was chosen to determine the effect of the size fractions distribution within the concrete. Unfortunately, the results did not show a difference in any of the levels of the variables tested and no conclusions could be made.

One reason why the concretes could not be distinguished from each other may have been the high cement factor of seven bags per cubic yard, employed throughout this investigation. This is supported by a recently reported freezing and thawing investigation on structural lightweight concrete (20). The investigation considered concrete of two levels of compressive strength containing 0, 33-1/3, 66-2/3 and 100 percent natural sand replacing the lightweight fines. It was concluded that nondurable concrete made with all lightweight fines could be made highly durable with partial or complete replacement of the fines with natural sand or by increasing the cement content and corresponding compressive strength.

The rating procedure, being of subjective nature, may have been another reason why the results of the scaling test showed no difference in the levels of the variables tested. It is very hard to put numbers on a deterioration test of this kind. Any rating procedure is entirely arbitrary and for this reason the specimens were rated by three observers, each using a different procedure. The results of the three observers were similar on the whole but for a few individual specimens, the rating varied by as much as four units.

Summary of Results

This study was restricted to two lightweight fine aggregates, hence, the findings can be applied only to lightweight fine aggregates of similar properties.

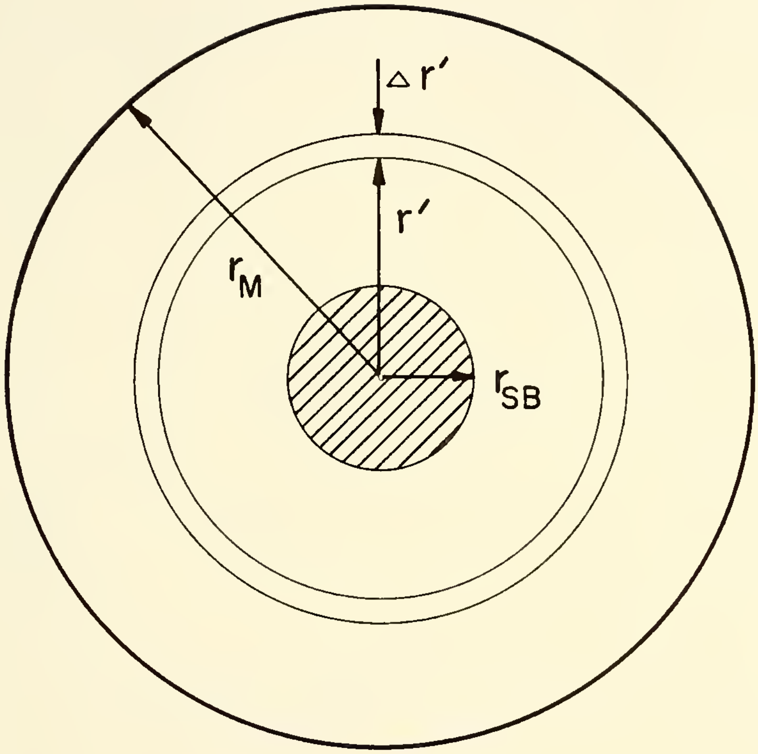
1. Neither of the two lightweight fine aggregates produced concretes as resistant to scaling as the control concrete.
2. An appreciable difference in scaling resistance was found between the two lightweight fine aggregate concretes. The noncoated aggregate produced the better scaling resistance concrete.
3. The noncoated aggregate had a lower porosity and a smaller amount of pores less than 5 microns than the coated aggregate.

"SOLID BUBBLE" HYPOTHESIS

It was mentioned in the introduction that if a conventional air-entrained bubble could be replaced by a "solid bubble" the problems of obtaining the proper amount and distribution of these bubbles could easily be solved.

Assuming that a sufficient quantity of these "solid bubbles" is present in the paste to protect the concrete as would regular air bubbles, the "solid bubble" theory is presented. Figure 31 shows a hypothetical cross-section through a "solid bubble" and its "sphere of influence".

Let r_M define the "sphere of influence" as presented by the hypothetical cross-section through a single "solid bubble" of radius r_{SB}. The paste is assumed to be composed of tiny spherical shells of thickness r' surrounding the "solid bubble". For the critical case the paste is assumed to have saturated capillary pores of freezable water. When freezing occurs, water will be expelled from the paste with a magnitude of approximately nine percent of the volume of



r_{SB} = RADIUS OF "SOLID BUBBLE"

r_M = "SPHERE OF INFLUENCE"

r' = ANY RADIUS

FIGURE 31 CROSS-SECTION THROUGH A "SOLID BUBBLE" AND ITS "SPHERE OF INFLUENCE"

the capillary pores. It is assumed that this water which is expelled from these spherical shells by the advancing ice will go into the "solid bubble" as it would if it were an air bubble. Therefore, if the "useful" porosity of the "solid bubble" is larger than or equal to the volume of the water expelled within its "sphere of influence" rupture of the paste will not occur.

The definition of "useful" porosity, ρ , is that volume of water the "solid bubble" would accept from the paste upon freezing. The derivation of an expression relating the "solid bubble" and its "sphere of influence" is presented below.

Let

$$\Delta v = 9\% \, C_p \left[4\pi (r')^2 \Delta r'\right]$$

where

Δv = the increment of water expelled from the paste

C_p = amount of capillary pores by volume of the paste

then

$$dv = \left[.09 C_p \, 4\pi\right] r'^2 \, dr$$

and

$$V = \int_{r_{SB}}^{r_M} \left[.09 \, C_p \, 4\pi\right] r'^2 \, dr$$

$$V = \left[.09\, C_p\, \frac{4}{3}\pi\right]\left[r_M^3 - r_{SB}^3\right]$$

where

$$V = \text{total water expelled from the paste.}$$

Let

$$V = \rho \times V_{SB}$$

where

$$\rho = \text{useful porosity of the "solid bubble"}$$

$$V_{SB} = \text{volume of the "solid bubble"}.$$

Then

$$V = \left[.09\, C_p\, \frac{4}{3}\pi\right]\left[r_M^3 - r_{SB}^3\right] = \rho\left[\frac{4}{3}\pi\, r_{SB}^3\right]$$

Solving for r_M

$$r_M^3 = r_{SB}^3\,(1 + K)$$

$$r_M = r_{SB}\,(1 + K)^{1/3}$$

where

$$K = \frac{\rho}{.09\, C_p}$$

It is now possible, knowing the percent capillary pores, the "useful" porosity and the radius of the "solid bubble", to calculate a critical spacing factor for the "solid bubble" to provide protection of the paste during freezing.

The spacing factor required to provide protection of the paste for conventional air-entrainment is around .01 of an inch or less (21).

The spacing factor is define as one half the maximum distance from any point in the cement paste from the periphey of an aggregate particle. Therefore, the spacing factor of the "solid bubble" can be found by using the following relation,

$$\text{Spacing factor} = 2(r_M - r_{SB}) .$$

A relationship between the spacing factor of a theoretical aggregate and its size fraction was found. A useful porosity of five percent was assumed for the theoretical aggregate. The amount of capillary pores of the paste was assumed to be 0.25. A maximum allowable spacing factor of 0.01 of an inch was assumed.

Figure 32 shows the spacing factor of the aggregate particles in inches versus its size fractions. If the aggregate particles act as "solid bubbles" the size fractions smaller than the No. 30 size are capable of protecting the paste if they are properly distributed.

The "solid bubble" hypothesis could not be proven. The two lightweight aggregate concretes were not as durable as the control sand concrete. This hypothesis could have been proven if the lightweight aggregate concretes had shown a

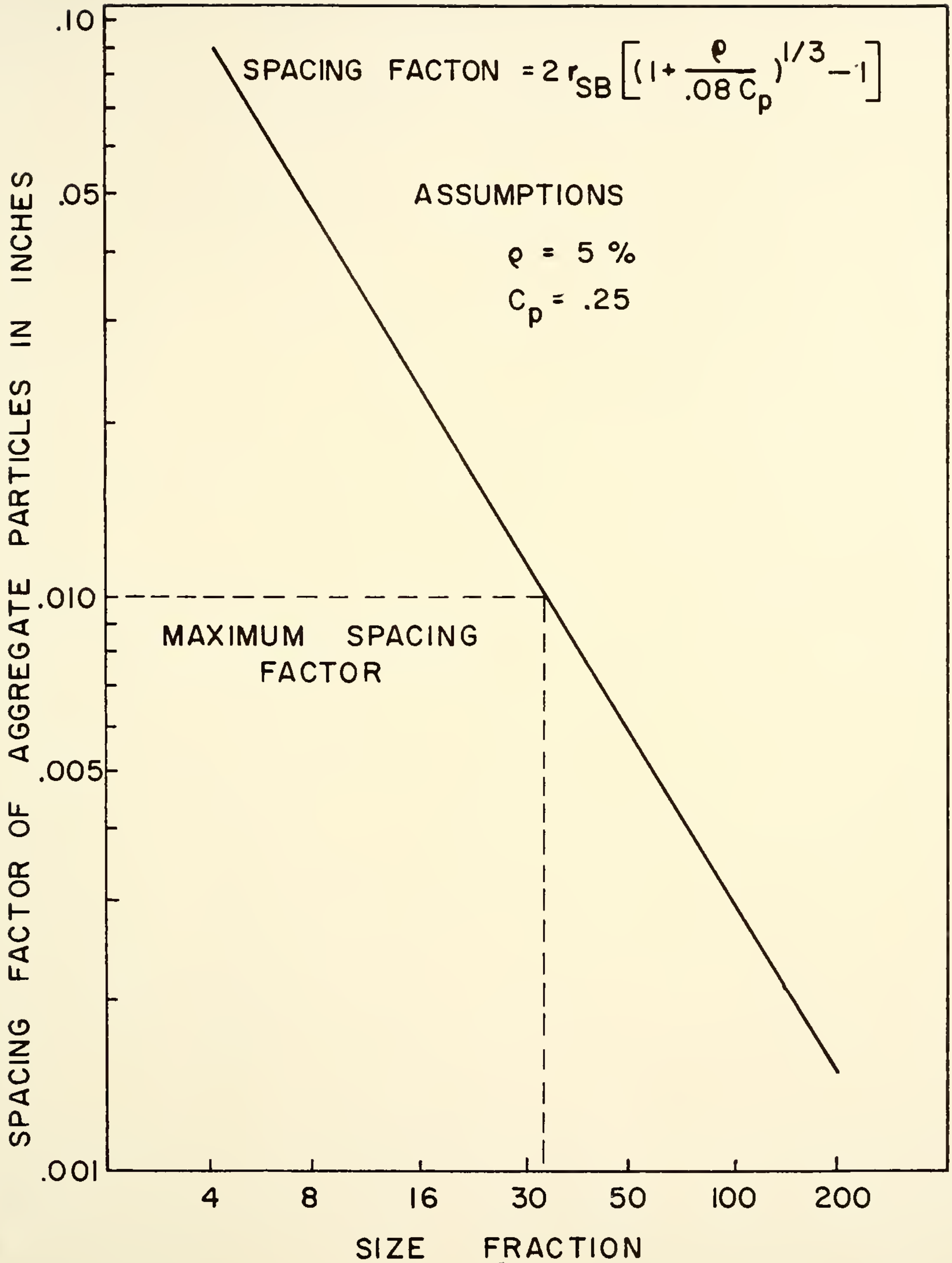


FIGURE 32 SPACING FACTOR OF AGGREGATE PARTICLES IN INCHES VS. SIZE FRACTION

better freeze-thaw scaling resistance than the control sand concrete.

CONCLUSIONS

Based on the results of this investigation, the following conclusions can be made.

1. The use of expanded shale fine aggregates substituted for the conventional fines in concrete does not improve the scaling resistance of concrete.
2. The expanded shale fines do not fulfill the requirements of the "solid bubble" hypothesis. However, this does not preclude successful use of some other type of "solid bubble" fine aggregate.
3. The freeze-thaw test, ASTM Designation: C 291-61T, The Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water, does not evaluate the potential scaling resistance of concrete.

PROPOSED RESEARCH

In view of this study, the following suggestions for the proposed research are presented.

1. Isolate the size fractions of the noncoated aggregate to determine which has the greatest effect on the durability of concrete.

2. Test the durability of concrete made with other lightweight aggregates.

3. Devise a test or method to determine the absorption of lightweight fine aggregates so the water/cement ratio may be controlled more precisely.

4. Use a lower cement factor to determine the effects of the lightweight aggregates.

5. Investigate the nature of the pores in more detail.

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APPENDICES

APPENDIX I

EXPERIMENTAL DESIGN

The experiment was designed with four variables, three with four levels and one with two levels. The design was a fractional factorial. A general description of the model is presented in Table A1. A complete description of the statistical design can be found in reference 22.

TABLE A1

MODEL DESCRIPTION

FACTOR	DESCRIPTION	NO. OF LEVELS
A	Lightweight Aggregate	2
B	Gradation	4
C	Water/Cement Ratio	4
D	Volume of Expanded Sand	4

The necessary assumptions for a model of this type are:

1) three factor interactions and higher are equal to zero;

2) experimental work was conducted under the same laboratory conditions and with no variations from day to day.

There are 2 x 4 x 4 x4= 128 combinations for the above proposed model. It was decided to use a fractional factorial design. One-half replication was employed with eight repeats chosen randomly from the 64 combinations. Thus, a total of seventy-two treatment combinations were produced for the experiment. The additional observations, or repeats, were utilized to estimate the experimental error, but were not used in estimating the effects of the treatments.

FACTOR AND PSEUDO FACTORS

The pseudo factors listed in Table A2 were used to determine which treatment combinations were to be run.

TABLE A2

FACTORS AND PSEUDO FACTORS

FACTOR	PSEUDO FACTOR	LEVELS
A	A	2
B	B_1	2
	B_2	2
C	C_1	2
	C_2	2
D	D_1	2
	D_2	2

Confounding and Aliases

Since only half of the treatment combinations were run, many effects could not be separated. These confounded effects were determined by using the defining contrast, I and multiplication mod 2. See Table A3 for some of the confounding and aliases. The defining contrast was chosen by the statistical consultant and the investigator in such a way that the information obtained from the data could be used most effectively.

TABLE A3

CONFOUNDING AND ALIASES

I	$= AB_1B_2C_1C_2D_1D_2$
A	$= B_1B_2C_1C_2D_1D_2$
AB_1	$= B_2C_1C_2D_1D_2$
AB_1B_2	$= C_1C_2D_1D_2$
AC_1C_2	$= B_1B_2D_1D_2$
AD_1D_2	$= B_1B_2C_1C_2$
$B_1B_2C_1C_2$	$= AD_1D_2$

Analysis of Variance

The final analysis of variance table is listed in Table A4 with some of the pseudo factor components listed at the side.

Treatment Combinations

To choose the treatment combinations only these combinations which satisfied

$$X_1 + X_2 + X_3 + X_4 + X_5 + X_6 + X_7 = 0 \bmod 2$$

where

$X_i = 1$ if the high level of factor i was used

$X_i = 0$ if the low level of factor i was used

were used. This equation came from the defining contrast.

Levels, Factors and Pseudo Factors

Table A5 shows the relation between the levels, factors and pseudo factors. This relation was used to produce the levels used in the experiment for the different combinations of variables. See Table A6 for the different level combinations of the treatment combinations.

TABLE A4

ANALYSIS OF VARIANCE TABLE

SOURCE		DEGREES OF FREEDOM	
Main Effects			
A		1	
B		3	
C		3	
D		3	
Two Factor Interactions			
AB_1		1	($AB_1B_2 = C_1C_2D_1D_2$
AB	2		due to confounding this information
AB_2		1	cannot be obtained)
AC_1		1	
AC	2		
AC_2		1	
AD		(3-1)=2	
BC		(9-1)=8	($B_1B_2C_1C_2 = AD_1D_2$)
BD		(9-1)=8	
CD		(9-1)=8	($C_1C_2D_1D_2 = AB_1B_2$)
Remainder		23	
Error (if used)		8	
Total		71	

TABLE A5

RELATION OF THE LEVELS, FACTORS AND PSEUDO FACTORS

Factor	A	B		C		D	
Pseudo Factor	A	B_1	B_2	C_1	C_2	D_1	D_2
Level of Factor							
1	0	0	0	0	0	0	0
2	1	0	1	1	0	0	1
3		1	0	0	1	1	0
4		1	1	1	1	1	1

TABLE A6

FACTOR LEVELS OF THE TREATMENT COMBINATIONS

NO.	TREATMENT COMBINATION	PSEUDO FACTOR							LEVEL OF FACTOR			
		X_1	X_2	X_3	X_4	X_5	X_6	X_7	A	B	C	D
		A	B_1	B_2	C_1	C_2	D_1	D_2				
1	(1)	0	0	0	0	0	0	0	1	1	1	1
2	ab_1	1	1	0	0	0	0	0	2	3	1	1
3	ab_2	1	0	1	0	0	0	0	2	2	1	1
4	ac_1	1	0	0	1	0	0	0	2	1	3	1
5	ac_2	1	0	0	1	1	0	0	2	1	2	1
6	ad_1	1	0	0	0	0	1	0	2	1	1	3
7	ad_2	1	0	0	0	0	0	1	2	1	1	2
8	b_1b_2	0	1	1	0	0	0	0	1	4	1	1
9	b_1c_1	0	1	0	1	0	0	0	1	3	3	1
10	b_1c_2	0	1	0	0	1	0	0	1	3	2	1
11	b_1d_1	0	1	0	0	0	1	0	1	3	1	3
12	b_1d_2	0	1	0	0	0	0	1	1	3	1	2
13	b_2c_1	0	0	1	1	0	0	0	1	2	3	1
14	b_2c_2	0	0	1	0	1	0	0	1	2	2	1
15	b_2d_1	0	0	1	0	0	1	0	1	2	1	3
16	b_2d_2	0	0	1	0	0	0	1	1	2	1	2
17	c_1c_2	0	0	0	1	1	0	0	1	1	4	1
18	c_1d_1	0	0	0	1	0	1	0	1	1	3	3

TABLE A6 (Con'd.)

NO.	TREATMENT COMBINATION	PSEUDO FACTOR							LEVEL OF FACTOR			
		X_1	X_2	X_3	X_4	X_5	X_6	X_7	A	B	C	D
		A	B_1	B_2	C_1	C_2	D_1	D_2				
19	c_1d_2	0	0	0	1	0	0	1	1	1	3	2
20	c_2d_1	0	0	0	0	1	1	0	1	1	2	3
21	c_2d_2	0	0	0	0	1	0	1	1	1	2	2
22	d_1d_2	0	0	0	0	0	1	1	1	1	1	4
23	$ab_1b_2c_1$	1	1	1	1	0	0	0	2	4	3	1
24	$ab_1b_2c_2$	1	1	1	0	1	0	0	2	4	2	1
25	$ab_1b_2d_1$	1	1	1	0	0	1	0	2	4	1	3
26	$ab_1b_2d_2$	1	1	1	0	0	0	1	2	4	1	2
27	$ab_1c_1c_2$	1	1	0	1	1	0	0	2	3	4	1
28	$ab_1c_1d_1$	1	1	0	1	0	1	0	2	3	3	3
29	$ab_1c_1d_2$	1	1	0	1	0	0	1	2	3	3	2
30	$ab_1c_2d_1$	1	1	0	0	1	1	0	2	3	2	3
31	$ab_1c_2d_2$	1	1	0	0	1	0	1	2	3	2	2
32	$ab_1d_1d_2$	1	1	0	0	0	1	1	2	3	1	4
33	$ab_2c_1c_2$	1	0	1	1	1	0	0	2	2	4	1
34	$ab_2c_1d_1$	1	0	1	1	0	1	0	2	2	3	3
35	$ab_2c_1d_2$	1	0	1	1	0	0	1	2	2	3	2
36	$ab_2c_2d_1$	1	0	1	0	1	1	0	2	2	2	3
37	$ab_2c_2d_2$	1	0	1	0	1	0	1	2	2	2	2

TABLE A6 (Con'd.)

NO.	TREATMENT COMBINATION	PSEUDO FACTOR							LEVEL OF FACTOR			
		X_1	X_2	X_3	X_4	X_5	X_6	X_7	A	B	C	D
		A	B_1	B_2	C_1	C_2	D_1	D_2				
38	$ab_2d_1d_2$	1	0	1	0	0	1	1	2	2	1	4
39	$ac_1c_2d_1$	1	0	0	1	1	1	0	2	1	4	3
40	$ac_1c_2d_2$	1	0	0	1	1	0	1	2	1	4	2
41	$ac_1d_1d_2$	1	0	0	1	0	1	1	2	1	3	4
42	$ac_2d_1d_2$	1	0	0	0	1	1	1	2	1	2	4
43	$b_1b_2c_1c_2$	0	1	1	1	1	0	0	1	4	4	1
44	$b_1b_2c_1d_1$	0	1	1	1	1	0	0	1	4	3	3
45	$b_1b_2c_1d_2$	0	1	1	1	0	0	1	1	4	3	2
46	$b_1b_2c_2d_1$	0	1	1	0	1	1	0	1	4	2	3
47	$b_1b_2c_2d_2$	0	1	1	0	1	0	1	1	4	2	2
48	$b_1b_2d_1d_2$	0	1	1	0	0	1	1	1	4	1	4
49	$b_1c_1c_2d_1$	0	1	0	1	1	1	0	1	3	4	3
50	$b_1c_1c_1d_2$	0	1	0	1	1	0	1	1	3	4	2
51	$b_1c_1d_1d_2$	0	1	0	1	0	1	1	1	3	3	4
52	$b_1c_2d_1d_2$	0	1	0	0	1	1	1	1	3	2	4
53	$b_2c_1c_2d_1$	0	0	1	1	1	1	0	1	2	4	3
54	$b_2c_1c_2d_2$	0	0	1	1	1	0	1	1	2	4	2
55	$b_2c_1d_1d_2$	0	0	1	1	0	1	1	1	2	3	4
56	$b_2c_2d_1d_2$	0	0	1	0	1	1	1	1	2	2	4

TABLE A6 (Con'd.)

NO.	TREATMENT COMBINATION	PSEUDO FACTOR							LEVEL OF FACTOR			
		X_1	X_2	X_3	X_4	X_5	X_6	X_7	A	B	C	D
		A	B_1	B_2	C_1	C_2	D_1	D_2				
57	$c_1c_2d_1d_2$	0	0	0	1	1	1	1	1	1	4	4
58	$ab_1b_2c_1c_2d_1$	1	1	1	1	1	1	0	2	4	4	3
59	$ab_1b_2c_1c_2d_2$	1	1	1	1	1	0	1	2	4	4	2
60	$ab_1b_2c_1d_1d_2$	1	1	1	1	0	1	1	2	4	3	4
61	$ab_1b_2c_2d_1d_2$	1	1	1	0	1	1	1	2	4	2	4
62	$ab_1c_1c_2d_1d_2$	1	1	0	1	1	1	1	2	3	4	4
63	$ab_2c_1c_2d_1d_2$	1	0	1	1	1	1	1	2	2	4	4
64	$b_1b_2c_1c_2d_1d_2$	0	1	1	1	1	1	1	1	4	4	4
65	$ac_1d_1d_2$	1	0	0	1	0	1	1	2	1	3	4
66	c_1d_1	0	0	0	1	0	1	0	1	1	3	3
67	$ac_1c_2d_1$	1	0	0	1	1	1	0	2	1	4	3
68	$ab_1c_1c_2d_1d_2$	1	1	0	1	1	1	1	2	3	4	4
69	$ab_1b_2c_1$	1	1	1	1	0	0	0	2	4	3	1
70	$b_1b_2c_2d_2$	0	1	1	0	1	0	1	1	4	2	2
71	ab_1	1	1	0	0	0	0	0	2	3	1	1
72	b_2c_1	0	0	1	1	0	0	0	1	2	3	1

Sum of Squares Due to Error

For the calculation of the sum of squares (ss) due to error (E) the observations taken from the repeats were utilized. Let

O_{j1} = the first observed observation from the j^{th} treatment combination $(j=1,2,\ldots,64)$

and

O_{j2} = the second observed observation from the j^{th} treatment combination (from the repeats)

then

$$\sum_j \frac{(O_{j1} - O_{j2})^2}{2} = SSE$$

= sum of squares due to the error within the treatment combinations j.

It was possible to test in the analysis of variance whether the assumption of negligible three factor and higher interactions was true or not by testing against the error sum of squares (SSE). If this was found to be significant, the remainder sum of squares (R) provided a conservative test for testing the main effects and the effects of two factor interactions. If it was not significant, the error sum of squares (SSE) was used for the testing.

APPENDIX II

STATISTICAL ANALYSIS OF THE ℓ/s RATIO

The statistical analysis of the ℓ/s ratio is presented in Table A7 below. This analysis was achieved using Two-Way Unequal Cell-Size Analysis of Variance, a computer program obtained from the Statistical Section of the Computer Science Department of Purdue University.

TABLE A7

STATISTICAL ANALYSIS OF THE ℓ/s RATIO

Output of Statistical Analysis

ANOV of ℓ/s Ratio

A Levels

1 = Noncoated Aggregate

2 = Coated Aggregate

B Levels

1 = No. 4 size fraction

2 = No. 8 size fraction

3 = No. 16 size fraction

4 = No. 30 size fraction

5 = No. 50 size fraction

Means

Levels of A	Levels of B				
	1	2	3	4	5
1	1.6474	1.7040	1.7778	1.8427	1.7603
2	1.6600	1.5376	1.5503	1.6364	1.7936

Marginal Means of A

Level 1 $\bar{X} = 1.7529$

Level 2 $\bar{X} = 1.6324$

TABLE A7 (Cont'd.)

Analysis of Variance

Source	Sum of Squares	DF	Mean Square	F Ratio
A Main Effects	3.56958008	1	3.569580	11.95*
B Main Effects	4.12906647	4	1.032267	3.46
Error	314.24320984	1052	0.298710	--

*Significant at 5% α level.

APPENDIX III

EXPERIMENTAL DATA

The experimental data is presented in Table A8. The data represents the result of the freeze-thaw scaling and compressive tests for specimens 1 through 80.

TABLE A8

EXPERIMENTAL DATA

MIX NO.	FACTOR LEVEL A	B	C	D	DURABILITY FACTOR IN PERCENT	SCALING RATING BY OBSERVER 1	2	3	COMPRESSIVE STRENGTH psi
1	1	1	1	1	99.1	3	3	3	6924
2	2	3	1	1	92.3	5	5	5	9249
3	2	2	1	1	100.0	5	5	5	9054
4	2	1	3	1	97.3	3	4	3	6383
5	2	1	2	1	99.1	4	5	4	7739
6	2	1	1	3	97.3	5	5	4	8432
7	2	1	1	2	98.2	4	5	4	8509
8	1	4	1	1	100.0	4	4	3	7144
9	1	3	3	1	101.9	3	3	3	3890
10	1	3	2	1	100.0	3	3	3	4506
11	1	3	1	3	100.0	2	2	1	6183
12	1	3	1	2	100.0	3	3	3	6359
13	1	2	3	1	100.0	4	4	3	4555
14	1	2	2	1	99.0	2	2	2	4294
15	1	2	1	3	98.1	3	3	2	5708
16	1	2	1	2	100.0	4	4	2	6118
17	1	1	4	1	99.0	4	4	2	2986
18	1	1	3	3	99.0	4	4	2	3558
19	1	1	3	2	102.0	2	4	1	3579

TABLE A8 (Con'd.)

MIX NO.	FACTOR LEVEL A	B	C	D	DURABILITY FACTOR IN PERCENT	SCALING RATING BY OBSERVER 1	2	3	COMPRESSIVE STRENGTH psi
20	1	1	2	3	98.1	2	2	1	4944
21	1	1	2	2	101.1	3	2	1	5270
22	1	1	1	4	99.0	3	2	2	5984
23	2	4	3	1	98.1	4	4	3	6274
24	2	4	2	1	98.2	4	4	3	7369
25	2	4	1	3	93.9	5	5	4	9196
26	2	4	1	2	96.5	5	5	4	9196
27	2	3	4	1	99.1	3	3	3	5475
28	2	3	3	3	97.3	3	3	2	7374
29	2	3	3	2	99.1	4	4	2	6034
30	2	3	2	3	98.2	5	5	4	7979
31	2	3	2	2	100.0	4	4	3	8311
32	2	3	1	4	91.2	5	5	5	9457
33	2	2	4	1	99.0	3	2	2	4584
34	2	2	3	3	98.1	3	4	2	6423
35	2	2	3	2	99.1	3	2	1	6338
36	2	2	2	3	96.4	4	4	3	7774
37	2	2	2	2	96.4	3	2	2	7696
38	2	2	1	4	97.3	4	4	3	7484
39	2	1	4	3	98.1	4	3	3	5617

TABLE A8 (Con'd.)

MIX NO.	FACTOR LEVEL A	B	C	D	DURABILITY FACTOR IN PERCENT	SCALING RATING BY OBSERVER 1	2	3	COMPRESSIVE STRENGTH psi
40	2	1	4	2	100.0	3	3	2	5115
41	2	1	3	4	99.1	4	2	2	5503
42	2	1	2	4	100.0	4	4	3	5956
43	1	4	4	1	98.0	1	2	1	2539
44	1	4	3	3	100.0	2	1	1	3346
45	1	4	3	2	99.0	2	1	1	4464
46	1	4	2	3	99.0	4	4	2	5454
47	1	4	2	2	100.0	2	2	2	5875
48	1	4	1	4	99.0	3	2	2	6504
49	1	3	4	3	98.0	3	4	3	3604
50	1	3	4	2	97.1	1	1	1	4081
51	1	3	3	4	98.0	2	2	1	4669
52	1	3	2	4	100.0	2	2	1	5355
53	1	2	4	3	98.1	2	2	1	5333
54	1	2	4	2	96.1	2	1	1	3332
55	1	2	3	4	99.0	4	2	2	4421
56	1	2	2	4	100.0	4	4	3	4909
57	1	1	4	4	98.0	4	4	2	3310
58	2	4	4	3	99.0	4	4	1	6175
59	2	4	4	2	100.0	5	5	3	6331

TABLE A8 (Con'd.)

MIX NO.	FACTOR LEVEL A	B	C	D	DURABILITY FACTOR IN PERCENT	SCALING RATING BY OBSERVER 1	2	3	COMPRESSIVE STRENGTH psi
60	2	4	3	4	100.0	4	5	2	6713
61	2	4	2	4	100.0	5	5	4	8184
62	2	3	4	4	98.1	4	5	2	6479
63	2	2	4	4	97.2	3	2	2	5843
64	1	4	4	4	97.0	4	4	2	3148
65	2	1	3	4	99.1	3	5	1	6020
66	1	1	3	3	99.0	2	4	2	3756
67	2	1	4	3	98.8	2	3	1	5737
68	2	3	4	4	99.8	4	5	3	6515
69	2	4	3	1	99.1	4	4	3	7187
70	1	4	2	2	99.8	3	3	3	5772
71	2	3	1	1	99.3	5	5	5	8828
72	1	2	3	1	99.8	4	3	1	3544
73	3	1	1	4	99.8	4			6650
74	3	4	1	1	98.2	2			8350
75	3	1	1	1	100.9	2			7120
76	3	1	4	1	100.8	1			4280
77	3	4	1	4	100.2	1			7325
78	3	1	4	4	98.2	2			4190
79	3	4	4	1	98.1	1			3580

TABLE A8 (Con'd.)

MIX NO.	FACTOR LEVEL				DURABILITY FACTOR IN PERCENT	SCALING RATING BY OBSERVER			COMPRESSIVE STRENGTH psi
	A	B	C	D		1	2	3	
80	3	4	4	4	97.2	2			4070

APPENDIX IV

STATISTICAL ANALYSIS OF THE DURABILITY FACTOR

The experiment as outlined in Procedures was a one-half replication of a 2x4x4x4 factorial. For ease, this was analyzed as a one-half replication of a 2x2x2x2x2x2x2 = 2^7 factorial using pseudo factors. Zero observations were placed in empty cells and the complete factorial analyzed. The sums of squares for the fractional factorial are obtained by multiplying by two. The analysis was achieved using BMDO2V-Analysis of Variance for Factorial Design, a computer program obtained from the Statistical Section of the Computer Sciences Department of Purdue University.

Table A9 contains the actual output from the analysis of the durability factors. The analysis of variance for this factorial design was obtained by converting the 2^7 factorial back to the 2x4x4x4 factorial and multiplying the sums of squares by two because of the zero observation cells. The determinations of the sums of squares can be found in Table A10.

The next step was to test whether the assumption of negligible three factor and higher interactions is significant or not by testing against the error sums of squares. The sum of squares due to error (SSE), utilizing the data obtained from the repeats, was calculated as outlined in Appendix I. The calculation of the sums of squares due to error can be found in Table All.

It was now possible to test to see if three and four factor interactions were equal to zero.

$$\text{Total SS} - \text{Residual} = 310907.54297 - 310688.61719$$
$$= 218.92578$$

$$\frac{\text{Total SS} - \text{Residual}}{2} - \text{Total Sum M.S.}$$
$$= 218.92578 - 177.13742$$
$$= 41.78836$$

Hypothesis: Third and Fourth iteractions were not significant.

$F_{23,\ 8} = 3.12$ at the 5% α level

$$F = \frac{41.788}{26.725} = 1.565$$

$1.565 < 3.12$

Therefore, three and four factor interactions were not significant. The ANOV table can be found in Table A12 the factors being tested against the error sums of squares (SSE) at the

five percent α level.

It was concluded from the analysis of variance of the durability factor that there were no significant difference in the levels of any of the factors analyzed.

TABLE A9

OUTPUT FROM THE ANALYSIS OF THE DURABILITY FACTOR

Number of Variables	1
Number of Factors	7
Number of Replicates	1

Factor	No. of Levels
1	2
2	2
3	2
4	2
5	2
6	2
7	2

Grand Mean 49.26718

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Squares
1	1	11.88281	11.88281
2	1	0.81281	0.81281
3	1	0.08000	0.08000
4	1	1.01531	1.01531
5	1	0.55125	0.55125
6	1	4.20500	4.20500
7	1	0.60500	0.60500

TABLE A9 (Con'd.)

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Squares
12	1	1.32031	1.32031
13	1	1.28000	1.28000
14	1	10.01282	10.01282
15	1	12.25125	12.25125
16	1	0.00500	0.00500
17	1	1.28000	1.28000
23	1	2.53125	2.53125
24	1	1.01531	1.01531
25	1	3.92000	3.92000
26	1	0.03125	0.03125
27	1	0.18000	0.18000
34	1	0.10125	0.10125
35	1	2.47531	2.47531
36	1	0.57781	0.57781
37	1	0.22781	0.22781
45	1	11.76125	11.76125
46	1	0.02000	0.02000
47	1	0.98000	0.98000
56	1	0.69031	0.69031
57	1	0.00281	0.00281
67	1	0.00031	0.00031

TABLE A9 (Con'd.)

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Squares
123	1	1.53125	1.53125
124	1	4.13281	4.13281
125	1	5.44500	5.44500
126	1	0.03125	0.03125
127	1	2.64500	2.64500
134	1	0.10125	0.10125
135	1	2.36531	0.36531
136	1	0.47531	0.47531
137	1	0.00781	0.00781
145	1	0.00125	0.00125
146	1	0.02000	0.02000
147	1	2.00000	2.00000
156	1	0.30031	0.30031
157	1	0.11281	0.11281
167	1	0.07031	0.07031
234	1	0.01125	0.01125
235	1	0.26281	0.26281
236	1	0.19531	0.19531
237	1	3.99031	3.99031
245	1	3.25125	3.25125
246	1	0.00000	0.00000

TABLE A9 (Con'd.)

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Squares
247	1	0.21125	0.21125
256	1	0.00281	0.00281
257	1	0.30031	0.30031
267	1	1.16281	1.16281
345	1	1.24031	1.24031
346	1	0.69031	0.69031
347	1	0.11281	0.11281
356	1	0.21125	0.21125
357	1	0.12500	0.12500
367	1	4.65125	4.65125
456	1	0.38281	0.33281
457	1	2.36531	2.36531
467	1	0.03781	0.03781
567	1	1.20125	1.20125
1234	1	1.20125	1.20125
1235	1	0.03781	0.03781
1236	1	2.36531	2.36531
1237	1	0.38281	0.38281
1245	1	4.65125	5.65125
1246	1	0.12500	0.12500
1247	1	0.21125	0.21125

TABLE A9 (Con'd.)

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Squares
1256	1	0.11281	0.11281
1257	1	0.69031	0.68031
1267	1	1.24031	1.24031
1345	1	1.16281	1.16281
1346	1	0.30031	0.30031
1347	1	0.00281	0.00281
1356	1	0.21125	0.21125
1357	1	0.00000	0.00000
1367	1	3.25125	3.25125
1456	1	3.99031	3.99031
1457	1	0.19531	0.19531
1467	1	0.26281	0.26281
1567	1	0.01125	0.01125
2345	1	0.07031	0.07031
2346	1	0.11281	0.11281
2347	1	0.30031	0.30031
2356	1	2.00000	2.00000
2357	1	0.02000	0.02000
2367	1	0.00125	0.00125
2456	1	0.00781	0.00781
2457	1	0.47531	0.47531

TABLE A9 (Con'd.)

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Squares
2467	1	2.36531	2.36531
2567	1	0.10125	0.10125
3456	1	2.64599	2.64500
3457	1	0.03125	0.03125
3467	1	5.44500	5.44500
3567	1	4.13281	4.13281
4567	1	1.53125	1.53125
12345	1	0.00031	0.00031
12346	1	0.00281	0.00281
12347	1	0.69031	0.69031
12356	1	0.98000	0.98000
12357	1	0.02000	0.02000
12367	1	11.76125	11.76125
12456	1	0.22781	0.22781
12457	1	0.57782	0.57782
12467	1	2.47531	2.47531
12567	1	0.10125	0.10125
13456	1	0.18000	0.18000
13457	1	0.03125	0.03125
13467	1	3.92000	3.92000
13567	1	1.01532	1.01532

TABLE A9 (Con'd.)

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Squares
14567	1	2.53125	2.53125
23456	1	1.28000	1.28000
23457	1	0.00500	0.00500
23467	1	12.25125	12.25125
23567	1	10.01281	10.01281
24567	1	1.28000	1.28000
34567	1	1.32031	1.32031
123456	1	0.60500	0.60500
123457	1	4.20500	4.20500
123467	1	0.55125	0.55125
123567	1	1.01531	1.01531
124567	1	0.08000	0.08000
134567	1	0.81282	0.81282
234567	1	11.88282	11.88282
RESIDUAL	1	310,688.61719	310,688.61719
TOTAL	127	310,907.54297	

TABLE A10

DETERMINATION OF THE SUMS OF SQUARES

Factor and Factor Interaction	Source of Variation (Pseudo Factors)	Mean Squares and Sum of Mean Squares	Sums of Mean Squares x 2
A	1	11.88281	23.76562
	2	0.81281	
B	3	0.08000	6.84812
	23	2.53125	
		3.42406	
	4	1.01531	
C	5	0.55125	26.65562
	45	11.76125	
		13.32781	
	6	4.20500	
D	7	0.60500	9.62062
	67	0.00031	
		4.81031	
	12	1.32031	
AB	13	1.28000	5.20062
		2.60031	
	14	10.01282	
AC	15	12.25125	44.52814
		22.26407	
	16	0.00500	
AD	17	1.28000	2.57000
		1.28500	
	24	1.01531	
	25	3.92000	
	245	3.25125	
BC	34	0.10125	24.55498
	35	2.47531	
	345	1.24031	
	234	0.01125	
	235	0.26281	
		12.27749	

TABLE A10 (Con'd.)

Factor and Factor Interaction	Source of Variation (Pseudo Factors)	Mean Squares and Sum of Mean Squares	Sums of Mean Squares x 2
	26	0.03125	
	27	0.18000	
	267	1.16281	
	36	0.57781	
BD	37	0.22781	22.03310
	367	4.65125	
	236	0.19531	
	237	3.99031	
		11.01655	
	46	0.02000	
	47	0.98000	
	467	0.03781	
	56	0.69031	
CD	57	0.00281	11.36060
	567	1.20125	
	456	0.38281	
	457	2.36531	
		5.68030	
		Total	177.13742

TABLE A11

CALCULATION OF THE SUMS OF SQUARES DUE TO ERROR

0_{j1}'s		0_{j2}'s		
Specimen No.	Durability Factor	Specimen No.	Durability Factor	$(0_{j1} - 0_{j2})^2/2$
41	99.1	65	99.1	0
18	99.0	66	99.0	0
39	98.1	67	98.8	0.245
62	98.1	68	99.1	1.440
23	98.1	69	99.1	0.500
47	100.0	70	99.8	0.020
02	92.3	71	99.3	24.500
13	100.0	72	99.8	0.020

$$\sum_j (0_{j1} - 0_{j2})^2/2 = SS_{error} = 26.725$$

TABLE A12

ANOV TABLE

Source	DF	MS	MS/SS_{error}	$F_{0.05;\nu 1\,\nu 2}$
A	1	23.76562	0.89	5.32
B	3	6.84812	--	4.07
C	3	26.65562	[illegible]	4.07
D	3	9.62062	--	4.07
AB	2	5.20062	--	4.46
AC	2	44.52814	1.67	4.46
AD	2	2.57000	--	4.46
BC	8	24.55498	.92	3.44
BD	8	22.03310	.84	3.44
CD	8	11.36060	--	3.44
REMAINDER	23	41.78836	1.57	3.12
	63			